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# OUTLINES OF EXPERIMENTAL CHEMISTRY

LUDLAM & PRESTON

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# OUTLINES OF EXPERIMENTAL CHEMISTRY

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PRELIMINARY EDITION

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# PREFACE

## TO PRELIMINARY EDITION

The course of practical work contained in this little volume makes no great pretensions to originality on the one hand, or to completeness on the other. It is printed in its present form as a matter of convenience, to bridge over a transition stage in the teaching of practical chemistry at Clifton, and it is issued for use in Clifton only, and much of it has been already worked through by boys using cyclostyled copies.

In Part I. the instructions given are of the very scantiest, and are omitted altogether in Section VI., as the boys at this stage work all together, and the teacher gives all the information necessary. The only reason for having instructions printed is for the case of the few boys at the commencement of the term who may be behind the other members of the class, and have to catch up in the first few lessons.

The later instructions are purposely meagre. They leave some absolute necessity for the boy to think, and to rely on the knowledge and experience which he has been accumulating. The boys work independently of one another; as soon as their notes for one experiment have been approved by the master they may start the next experiment. In the boys' notebook is a copy of the list of experiments, and as each boy completes an experiment he crosses off the number of it on the list. Thus a master knows at a glance what work the boy has done. The master uses his judgment as to what experiments shall be omitted and what others included. There is no rigidity at all.

E. B. L.
H. P.

*September*, 1909.

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# OUTLINES OF EXPERIMENTAL CHEMISTRY

## SCHEME OF WORK—PART I.

### SECTION I.—Use of the Balance.

Exp. 1. Draw the various parts of the balance.

Exp. 2. Examine the weights and check them against each other.

Exp. 3. Arrange weights on pan according to instructions.

Exp. 4. Weigh metal discs.

Exp. 5. Weigh a crucible first hot and then cold, and notice if there is any difference.

Exp. 6. Find the weight of 1 cm. of fine wire, and also the length of wire which would weigh 1 gram.

Exp. 7. Find the weight of 1 cm. of glass tube and of glass rod.

Exp. 8. Find the gain or loss in weight on heating—

(*a*) Dry sand.
(*b*) Zinc carbonate.
(*c*) Magnesium.

### SECTION II.—Volumes.

Exp. 9. Find the volume of a test-tube by weighing.

Exp. 10. Find the volume of a flask by weighing.

Exp. 11. Use the graduated cylinder, checking the above.

Exp. 12. Find the volume of a stopper by dropping it into a cylinder.

Exp. 13. Find the volume of sand by dropping it into a cylinder.

Exp. 14.—Calibrate a pipette (*a*) blowing out the last drop; (*b*) leaving the last drop in.

Exp. 15. Calibrate a burette.

Exp. 16. Calibrate cylinder by pipette.

Exp. 17. Find the weight of 10 c.c. of salt solution and other liquids.

Exp. 17A. *If time permits,* find the densities of lumps of metal, etc.

## SECTION III.—Fitting up Apparatus.

Exp. 18. Draw a Bunsen burner, showing how it works.

Exp. 19. Draw out a jet in the Bunsen flame.

Exp. 20. Bend right angles, etc.

Exp. 21. Cut a clean edge (on glass) and round off.

Exp. 22. Make a model Bunsen.

Exp. 23. Bore one hole in a cork to fit a tube.

Exp. 24. Bore two holes in a cork to fit two tubes.

Exp. 25. Fit up an aspirator.

Exp. 26. *If time permits*—
- (*a*) Seal up a tube.
- (*b*) Bend U-tube—combination bends.
- (*c*) Join rod and tube.
- (*d*) Draw out capillary tubes.
- (*e*) Make bulbs.
- (*f*) Make a 5 c.c. pipette.

## SECTION IV.—Solubilities.

Exp. 27. Evaporate tap-water, etc.

Exp. 28. Find total solids in sea-water.

Exp. 29. Filter and repeat Exp. 28.

Exp. 30. Distil and repeat Exp. 28.

Exp. 31. Find if calcium sulphate dissolves in water. Find if the given substances dissolve in (*a*) hot water; (*b*) in cold water.

Exp. 32. Make up a saturated solution of salt, and find the amount of salt in solution, using the burner to evaporate.

Exp. 33. Same as Exp. 32, using the sand-bath.

Exp. 34. Same as Exp. 32, using the water-bath.

Exp. 35. Find the solubility of nitre at 15°.

Exp. 36. Find the solubility of alum at 15°.

Exp. 37. Plot the solubility curve.

Exp. 38. Separate salt and sand quantitatively.

Exp. 39. Separate salt and nitre, and obtain a good yield of each.

Exp. 40. Find the amount of dissolved air in a litre of tap-water.

Exp. 40a.—*If time permits*—

(*a*) Exp. 35 for copper sulphate at 15°.
(*b*) Separate chalk and salt, nitre and carbon, sand and sugar.
(*c*) Dissolve carbon dioxide in "flat" soda water.
(*d*) Determine total solids in other natural waters.
(*e*) Determine hardness, using soap solution.

## SECTION V.—Crystallisation.

Exp. 41. Make nitre crystals (*a*) cooling quickly; (*b*) slowly.

Exp. 42. Make crystals of alum.

Exp. 43. Make crystals of copper sulphate.

Exp. 44. Find water of crystallisation in copper sulphate.

Exp. 44a. *If time permits*—

(*a*) Crystals of sulphur from carbon disulphide.
(*b*) Naphthalene from alcohol.
(*c*) Potassium dichromate, zinc sulphate, chrome alum, ferrous ammonium sulphate.
(*d*) Find if $x$, $y$, and $z$ contain water of crystallisation.

## SECTION VI.—Physical and Chemical Change.

Exp. 45. Determine a melting point.

Exp. 46. Determine a melting point by cooling.

Exp. 47. Determine a boiling point.

Exp. 48. Find the action of heat on salt, limestone, potash, soda, copper sulphate, copper carbonate, ferrous sulphate, sulphur, ammonium chloride, lead nitrate, ammonium nitrate, ammonium carbonate.

Exp. 49. Action of water on the above substances before and after heating.

Exp. 49a. *If time permits,* find the action of heat and water upon copper nitrate, lead chloride, potassium tartrate, turpentine.

Exp. 50. Mix iron and sulphur—mixture and compound.

Exp. 51. Grind together mercury and iodine.

Exp. 52. Formation of oxides — magnesium, lead, copper.

Exp. 53. Reduction of oxides on carbon with blowpipe —lead, silver, bismuth.

Exp. 53a. *If time permits,* reduce oxides of copper, tin, antimony, and iron.

Exp. 54. Replace tin, copper, and lead in solution.

Exp. 54a. *If time permits,* replace silver by copper.

Exp. 55. Double decomposition—prepare calcium carbonate, lead chloride, lead sulphide, lead chromate, ferric hydroxide, chromium hydroxide, barium sulphate.

Exp. 55a. *If time permits,* borax bead reductions and oxidations of iron and manganese.

# SCHEME OF WORK—PART II.

## SECTION I.—Combustion.

Exp. 1. Find if metals gain or lose in weight when heated. Use magnesium, tin, lead, copper.

Exp. 2. Find how much 100 gms. of magnesium gains in weight on burning.

Exp. 3. Find whether phosphorus gains in weight on burning.

Exp. 4. Find whether a candle gains in weight on burning.

Exp. 5. Study the action of heat on mercuric oxide.

Exp. 6. Find whether the following bodies give off oxygen when heated in test-tubes: potassium nitrate, barium peroxide, copper oxide, calcium oxide, potassium permanganate, manganese dioxide, potassium chlorate. They all contain oxygen.

Exp. 7. Preparation of oxygen.

Exp. 8a. Find the weight of oxygen driven off from potassium chlorate by heat.

Exp. 8b. Find the volume of oxygen driven off.

Exp. 8c. Dissolve the residue in water and add silver nitrate. Do the same with some potassium chlorate and compare.

Exp. 9. Prepare nitrogen by passing air over heated copper.

Exp. 10. Measure the volume of nitrogen in air.

Exp. 11. Study the formation of rust.

Exp. 12. Obtain metals by reduction.

## SECTION II.—Common Acids and Alkalies.

Exp. 13. Examine the effect of heat on ferrous sulphate crystals.

Exp. 14. Experiments with sulphuric acid.

Exp. 15. Prepare hydrochloric acid.

Exp. 16. Prepare nitric acid.

Exp. 17. Preparation and properties of carbonic acid.

Exp. 17a. Find the loss of carbon dioxide when 100 gms. of calcium carbonate is dissolved by hydrochloric acid.

Exp. 17b. Find the loss in weight of 100 gms. of calcium carbonate when strongly heated.

Exp. 17c. Find the volume of carbon dioxide evolved from 100 gms. of calcium carbonate.

Exp. 18. Examine the action of sulphur and iron at a red-heat.

### *Alkalies.*

Exp. 19. To note changes which take place upon adding water drop by drop to the residue from Exp. 17c.

Exp. 20. Examine soda and potash.

Exp. 21. Prepare sodium hydrate.

Exp. 22. Prepare ammonia gas.

## SECTION III.—Salts.

Exp. 23. Prepare sodium chloride, using sodium carbonate and hydrochloric acid.

Exp. 24. Prepare sodium chloride, using sodium hydrate and hydrochloric acid.

Exp. 25. Prepare ammonium chloride, using ammonium hydrate and hydrochloric acid.

Exp. 26. Prepare calcium chloride, using lime and hydrochloric acid.

Compare all these substances, Exp. 23–26, as to appearance, taste, action of heat.

Exp. 27. Prepare potassium nitrate, using potash and nitric acid.

Exp. 28. Prepare sodium nitrate, using soda and nitric acid.

Exp. 29. Prepare ammonium nitrate, using ammonium hydrate and nitric acid.

Exp. 30. Prepare calcium nitrate, using lime and nitric acid.

Compare Exp. 27–30 as before.

Exp. 31. Repeat Exp. 23, but take dilute sulphuric acid and neutralise still more carefully than in the previous cases.

Exp. 32. Add lime-water to dilute sulphuric acid containing litmus until neutral. Describe fully what happens.

While the solutions obtained above are evaporating, compare the strengths of the solutions given.

| | *A* | *B* |
|---|---|---|
| Exp. 33. | Sulphuric acid | Sodium hydrate |
| Exp. 34. | Hydrochloric acid | Ammonia |
| Exp. 35. | Nitric acid | Lime-water |

### SECTION IV.—The Action of Dilute Acids on Metals.

Using the metals zinc, iron, magnesium, lead, copper, tin.

Exp. 36. Using sulphuric acid.

Exp. 37. Using hydrochloric acid.

Exp. 38. Using nitric acid.

Exp. 39. Preparation of hydrogen.

Exp. 40. To find the weight of oxygen that combines with one gram of hydrogen.

Exp. 41. To collect the products formed when hydrogen is burnt.

Exp. 42. The decomposition of water by sodium and calcium.

Exp. 43. Prepare hydrogen by passing steam over red-hot iron.

### SECTION V.—The Action of Strong Acids on Metals.

Repeat Exp. 36, 37, and 38, but now use strong acids.

Exp. 44. Using hydrochloric acid.

Exp. 45. Using sulphuric acid.

Exp. 46. Prepare sulphur dioxide.

Exp. 47. Action of strong nitric acid on metals.

Exp. 48. Prepare nitric oxide.

Exp. 49. Investigate the action of dilute nitric acid [1 : 10] on zinc.

Exp. 50. Prepare nitrous oxide.

### SECTION VI.—Equivalent Weights.

Exp. 51. Find the equivalent weight of magnesium by collecting the hydrogen evolved when it dissolves in hydrochloric acid.

Exp. 52. Find the equivalent weight of magnesium by igniting it in air.

Exp. 53. Find the equivalent weight of zinc by collecting the water displaced by the hydrogen produced when the metal is dissolved in an acid.

Exp. 54. Find the equivalent weight of aluminium by collecting the hydrogen evolved when the metal dissolves in sodium hydrate.

Exp. 55. Find the equivalent weight of copper by reduction of its oxide by hydrogen.

Exp. 56. Find the equivalent weight of copper by replacement.

Exp. 57. Find the equivalent weight of zinc by replacement.

### SECTION VII.—Experiments on the Halogens.

Exp. 58. Prepare hydrochloric acid. [No. 15.]

Exp. 59. Action of oxidising agents on hydrochloric acid.

Exp. 60. Prepare chlorine.

Exp. 61. Examine the action of strong sulphuric acid on potassium bromide.

Exp. 62A. Study the action of oxidising agents on hydrobromic acid.

Exp. 62B. Study the action of (*a*) ordinary dilute sulphuric acid, (*b*) strong sulphuric acid on potassium iodide.

Exp. 62C. Study the action of oxidising agents on hydriodic acid.

### SECTION VIII.—Volumetric Analysis.

Exp. 63. Study the behaviour of the indicators, litmus, methyl orange, phenol phthalein.

Exp. 64. Make a normal solution of sodium carbonate.

Exp. 65. Make a normal solution of sulphuric acid.

Exp. 66. Find the amount of sodium hydrate (in gms. per c.c.) in a given solution, using $N$ acid made in Exp. 65.

Exp. 67. Find the amount of hydrochloric acid in gms. per c.c. in a given solution, using the $N$ sodium carbonate made in Exp. 64.

Exp. 68. Find the weight of potassium carbonate in the given tube, using standard sulphuric acid [1 c.c. = 0·0061 gm. $H_2SO_4$].

Exp. 69. Find the percentage amount of pure hydrochloric acid in the commercial acid.

Exp. 70. Find the percentage amount of pure oxalic acid in the given crystals, using (*a*) litmus, (*b*) methyl orange, (*c*) phenol phthalein.

Exp. 71. Find the strength of the given *strong* ammonia solution in gms. per 100 c.c.

Exp. 72. Find the strength of the ammonium chloride solution (in gms. per litre) by boiling out the ammonia with standard alkali.

Exp. 73. Find the strength of the given ammonium chloride solution (in gms. per litre) by distilling the ammonia into standard acid.

Exp. 74. Find the proportion of sodium carbonate in washing-soda and deduce the formula of the salt.

Exp. 75. Find the basicity of the given acid whose molecular weight is 90.

Exp. 76. Find the percentage amount of barium chloride in 100 gms. of the given crystals.

Exp. 77. Find the amount of ammonium chloride in a given solution (in gms. per litre), using $\frac{N}{10}$ silver nitrate (*a*) without the aid of an indicator, (*b*) with the aid of an indicator.

Exp. 78. Find the strength of a barium chloride solution (in gms. per litre), using $\frac{N}{10}$ silver nitrate.

## SECTION IX.—Preparations.

Exp. 79. Prepare pure sodium chloride.

Exp. 80. Prepare the neutral and acid salts of sulphuric acid.

Exp. 81. Prepare sodium thiosulphate.

Exp. 82. Prepare sodium hydrogen carbonate from sodium carbonate.

Exp. 83. Prepare potassium nitrate from sodium nitrate and potassium carbonate.

Exp. 83a. Prepare potassium chlorate as in the manufacture.

Exp. 84. Prepare potash alum.

Exp. 85. Prepare pure ammonium chloride from gas liquor.

Exp. 86. Prepare metallic copper from azurite.

Exp. 87. Prepare crystals of copper sulphate from copper pyrites.

Exp. 88. Prepare 10 gms. of copper carbonate from the sulphate by precipitation with sodium carbonate.

Exp. 89. Prepare the nitrate from copper and convert it into the oxide.

Exp. 90. Prepare cuprous chloride from copper oxide, copper, and hydrochloric acid.

Exp. 91. Convert a 3*d.* piece [supplied by student] into silver chloride.

Exp. 92. Pass coal gas over silver chloride obtained from Exp. 91.

Exp. 93. To obtain silver by cupellation.

Exp. 94. Obtain the metal from galena.

Exp. 95. To obtain lead oxide from lead carbonate.

Exp. 96. Convert litharge into red lead.

Exp. 97. Convert red lead into lead peroxide.

Exp. 98. Prepare lead chloride.

Exp. 99. Prepare lead iodide.

Exp. 100. Obtain mercury from cinnabar.

Exp. 101. Prepare mercuric iodide.

Exp. 102. Prepare zinc oxide from the carbonate.

Exp. 103. Prepare zinc sulphate from the oxide.

Exp. 104. Prepare magnesium sulphate crystals from magnesite.

Exp. 105. Prepare ferrous ammonium sulphate, $FeSO_4(NH_4)_2SO_47H_2O$.

## SECTION X.—Atomic Theory.

Exp. 106. Find the density of air.

Exp. 107. Find the density of carbon dioxide.

Exp. 108. Find the density of hydrochloric acid.

Exp. 109. Find the density of ammonia.

Exp. 110. Compare the rates of diffusion.

Exp. 111. Find the composition of water by volume.

Exp. 112. Find the composition of hydrochloric acid by volume.

Exp. 113. Find the composition of carbon monoxide.

Exp. 114. Find the composition of ammonia by volume.

Exp. 115. Find the atomic weight of potassium, chlorine, silver.

Exp. 116. Find the vapour density by Victor Meyer's method.

Exp. 117. Find the molecular weight of Raoult's method.

Exp. 118. Find the molecular weight by Landsberger's method.

Exp. 119. Find the electro-chemical equivalent [Lupke, p. 38].

Exp. 119a. Find the specific heat of mercury.

## SECTION XI.—Special Volumetric Analysis.

Exp. 120. Find if the given substance is carbonate or bicarbonate of sodium.

Exp. 121. Find the weight of magnesium in the given tube.

Exp. 122. Find the percentage of pure calcium chloride in the given sample.

Exp. 123. Find the amount (in gms. per litre) of sodium and barium chlorides in a solution.

Exp. 124. Find the amount in gms. per litre of ammonium and sodium hydrates in a solution.

Exp. 125. Find the percentage amount of caustic soda and sodium carbonate in commercial caustic soda.

Exp. 126. Standardise a solution of potassium permanganate with ferrous-ammonium sulphate.

Exp. 127. Find the percentage of iron in a sample of iron wire.

Exp. 128. Find the percentage amount of iron present in a sample of spathic iron ore by means of standard permanganate.

Exp. 129. Find the percentage of iron in a sample of iron wire, and (*b*) to find the percentage of iron in spathic iron ore, using standard potassium bichromate.

Exp. 130. Make a deci-normal solution of iodine, and then standardise with a solution of sodium thiosulphate.

Exp. 131. Estimate the available chlorine in bleaching powder.

Exp. 132. Find the percentage of manganese dioxide [$MnO_2$] in a given sample.

Exp. 133. Find the amount of potassium iodide and chloride in a solution.

## INSTRUCTIONS.

The student must always make full notes of the experiments he performs, and draw diagrams of apparatus the first time he uses it.

In the work of Part I. the teacher will explain how the notes are to be taken.

The notes for each experiment should be started on a fresh page, headed with the date on which the experiment was started, its number and title.

Answers to Questions "A" must be written before commencing an experiment, and passed by the master.

Questions "B" must be answered and passed before commencing the next experiment.

In writing notes of an experiment in which numerical results have been obtained, it is better to write the description, omitting the actual figures, but these should be tabulated very clearly below the description.

### Typical Example.

Part I. No. 44. 26/5/09.

To find the Water of Crystallisation in Copper Sulphate.

**Description.**—I first weighed a crucible, and then placed in it some powdered copper sulphate, and weighed again. I now placed the crucible and its contents on a pipeclay triangle resting on a tripod, and heated by means of a Bunsen burner, until no further change appeared to take place. The copper sulphate, which had a blue colour to commence with, now turned white. I then allowed the crucible and contents to cool, and when cold weighed again.

**Results.**

*Before Heating.*

Grams.
Weight of crucible and copper sulphate = 17·72
Weight of crucible alone = 15·23

Weight of copper sulphate = 2·49

*After Heating.*

Grams.
Weight of crucible and copper sulphate = 16·82
Weight of crucible alone = 15·23

Weight of copper sulphate = 1·59

Weight of copper sulphate before heating = 2·49
Weight of copper sulphate after heating = 1·59

∴ Loss in weight = 0·90

2·49 gms. of copper sulphate have lost 0·90 gm.
∴ 100 gms. of copper sulphate will lose

$$\frac{100\times 0{\cdot}9}{2{\cdot}49}=\frac{90}{2{\cdot}49}=36{\cdot}14\%$$

The description should always be as full as possible, and the student should always be on the alert to note the colour, appearance, smell, etc., of all substances, and any changes which take place during the experiment. Although the instructions are given in the imperative mood, the actual notes should avoid using the same style, *e.g.* in the headings the notes should read—To find the volume, etc., although the book says—Find the volume, etc.

# PART I.

## SECTION I.—Use of the Balance.

### *Directions for weighing.*

Before beginning to weigh—

(1) See that the scale pans are clean;

(2) See that the beam swings evenly, and is resting properly on its knife edges.

(3) See that there are no weights missing from the box. [If the balance is not in adjustment or the scale pans are dirty, the fact should be reported AT ONCE to the master in charge.]

When weighing—

(1) No chemicals are to be placed on the scale pans; they should be placed for weighing in a *clean* dish whose weight is known.

(5) Place the object to be weighed on the left pan of the balance, and the weights on the right.

(6) Nothing is to be placed on or removed from either scale pan while the beam is swinging.

(7) Write down the weights on the scale pan in your rough note-book before removing them, and check each weight as you remove it.

(8) Never add up the weights on the scale pan in your head.

(9) On no account must you touch the weights with your hands.

Exp. 1. Draw the various parts of the balance.

The balance may be taken to pieces and the parts drawn separately and as simply as possible, *e.g.* the beam, stirrups, knife edges, pointer and scale, adjustment screws.

Exp. 2. Examine the weights and check them against each other.

Draw a plan of the box of weights, marking the position of each weight.

Exp. 3. Arrange weights on the pan according to instructions.

Pick out the proper weights to make a total of 5·5, 10·6, 8·45, 23·87, 49·79.

Exp. 4. Weigh metal discs.

Numbered pieces of metal are supplied of known weight.

Exp. 5. Weigh a crucible first hot and then cold, and notice if there is any difference.

Use a porcelain crucible.

Exp. 6. Find the weight of 1 cm. of fine wire, and also the length of wire which would weigh 1 gram.

Exp. 7. Find the weight of 1 cm. of glass tube, and of glass rod.

Exp. 8. Find the gain or loss on heating—

(*a*) Dry sand;
(*b*) Zinc carbonate;
(*c*) Magnesium;

Weigh an empty crucible, fill it to about half full of the substance, weigh again, heat strongly, allow to cool, and again weigh. Arrange the results in the style shown on p. 12.

## SECTION II.—Volumes.

Exp. 9. Find the volume of a test-tube by weighing.

Find the volume of a test-tube up to a mark by weighing.

Weigh the tube empty, fill it up to a mark with water, and weigh again.

Exp. 10. Find the volume of a flask by weighing.

Do the same as in the previous experiment, but using a flask.

Exp. 11. Use the graduated cylinder, checking the above.

Pour the contents of the flask into the cylinder and read the volume.

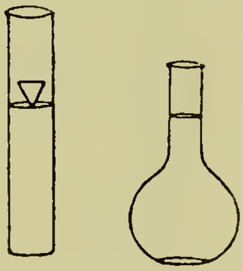

Fig. 1.

Exp. 12. Find the volume of a stopper by dropping it into a cylinder.

Weigh a stopper, slide it gently into a cylinder half full of water. The level of the water must be read exactly before and after dropping in the stopper.

Exp. 13. Find the volume of sand by dropping it into a cylinder.

Weigh 20 gms. of sand, and do the same as with the stopper.

Exp. 14. Calibrate a pipette (*a*) blowing out the last drop ; (*b*) leaving the last drop in.

Weigh a basin and run into it the water from a pipette. A drop will collect at the bottom of the pipette, but will not fall unless blown out. See which gives the best result, assuming 1 c.c. weighs 1 gram.

Exp. 15. Calibrate a burette.

Fill the burette with water to the mark 0. Run out 10 c.c. at a time into a weighed basin and tabulate the five weighings, so as to show the weight of each 10 c.c.

Exp. 16. Calibrate cylinder by pipette.

Run 20 c.c. at a time from a pipette into a 200-c.c. cylinder, read the level carefully each time, and tabulate the ten readings.

Exp. 17. Find the weight of 10 c.c. of salt solution and other liquids.

Exp. 17a. *If time permits,* find the densities of lumps of metal, etc.

## SECTION III.—Fitting up Apparatus.

(Instructions given orally and with demonstrations.)

## SECTION IV.—Solubilities.

Exp. 27. Evaporate tap-water, etc.

Place 5 c.c. of each kind of water on a watch-glass, evaporate off the water, using a sand-bath, describe and compare the residues.

Exp. 28. Find total solids in sea-water.

Take 20 c.c. of the water, run it into a weighed basin, evaporate to dryness and again weigh.

Exp. 31. Find if calcium sulphate dissolves in water.

Shake up some calcium sulphate with water for five minutes in a test-tube, filter and evaporate a few c.c. on a watch-glass.

Exp. 32. Make up a saturated solution of salt and find the amount of salt in solution, using the burner to evaporate.

In making up the saturated solution take care to have plenty of salt left after shaking for ten minutes. Then filter the solution. Weigh a basin, run in 20 c.c. of the filtered solution, weigh again, evaporate to dryness, and find the weight of the residue. Arrange the results as shown on p. 12, and calculate the weight of salt that 100 *gms. of water* have dissolved.

Exp. 35. Find the solubility of nitre at 15°.

Make up a saturated solution of nitre, using a thermometer. If the solution is too cold, it must be warmed.

Exp. 38. Separate salt and sand quantitatively.

Weigh out 10 gms. of the mixture, add to it 30 c.c. of hot water in a beaker. Stir for five minutes. Prepare two filter papers of equal weight, fold them together, and place in the funnel. Filter the solution, transfer all the sand to the filter paper, and wash it with water, using a wash-bottle. Measure the volume of the liquid, and take a definite fraction of it to evaporate down. So obtain the weight of salt. The sand and the filter papers are placed in an air oven to dry, and so obtain the weight of sand.

Exp. 39. Separate salt and nitre, and obtain a good yield of each.

Mix 20 gms. of nitre and 20 gms. of salt, dissolve as far as possible in 40 c.c. of boiling water, filter, and allow the filtrate to cool and crystallise. The crystals are then drained, dried, and weighed.

Exp. 40. Find the amount of dissolved air in a litre of tap-water.

Completely fill the flask and delivery tube with the water. Boil the water to drive out the air, and collect it in a test-tube standing over water.

## SECTION V.—Crystallisation.

Exp. 41. Make nitre crystals (*a*) cooling quickly; (*b*) slowly.

Take about 10 gms. of nitre, put half of it into one test-tube, the other half into another, and add about 10 c.c. of water. Heat until the nitre is dissolved. Allow a stream of cold water to run over one test-tube, and shake it. Put the other aside to cool slowly. Compare the crystals obtained in the two test-tubes.

Exp. 42. Make crystals of alum.

Dissolve about 20 gms. of alum in 100 gms. of water, filter, and put aside to cool slowly.

Exp. 43. Make crystals of copper sulphate.

Dissolve about 3 gms. of copper sulphate in 100 c.c. of water, filter, and put aside to cool slowly.

Exp. 44. Find the water of crystallisation in copper sulphate.

Grind up some pure copper sulphate, weigh a basin, add about 2 gms. of copper sulphate and weigh again. Heat gently until all the copper sulphate has become white. Reweigh when cold, and calculate the loss on heating 100 gms. of the copper sulphate.

(Tabulate results as in the example on p. 12.)

# PART II.

## SECTION I.—Combustion.

1. OBJECT.—To find whether metals gain or lose in weight when heated. Use magnesium, tin, lead, and copper.

*Questions A.*—(1) What apparatus do you require?
(2) How much of the metal?
(3) In what condition would you like the metal?
(4) How long do you propose to heat it?

*Questions B.*—(1) What changes took place during heating?
(2) What changes took place on cooling?

What conclusion do you draw from the experiment?

2. OBJECT.—To find out how much 100 gms. of magnesium gains in weight on heating.

*Questions A.*—(1) What apparatus will you select?
(2) How much magnesium will you take?
(3) How long will you heat?
(4) What precautions will you take?

Record your result in tabular form.

3. OBJECT.—To find whether phosphorus gains in weight on burning. A convenient apparatus can be made out of a test-tube (see diagram) by making a small hole in the bottom, and fitting it up as shown in the figure. The air is drawn over the phosphorus in a gentle stream, and the phosphorus will burn directly it is warmed by a Bunsen flame.

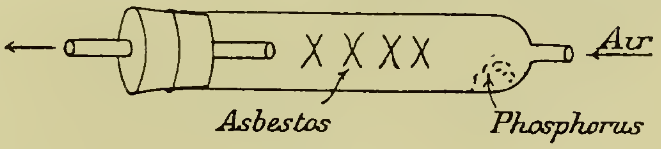

FIG. 2.

*Questions A.*—(1) How do you propose to carry out the experiment?
(2) What is the asbestos for?

(3) Why is a current of air needed?

(4) Why must the current be gentle?

(5) Why do you suppose a small hole is preferred for the air to enter by?

4. Object.—To find whether a candle gains in weight on burning. A lamp-glass is a suitable piece of apparatus to employ. The candle can be fixed as shown in the figure. The products of combustion are caught by the caustic potash and calcium chloride.

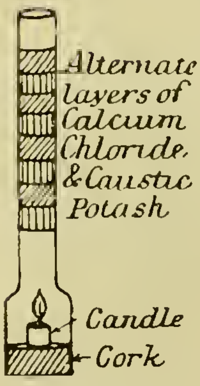

Fig. 3.

*Questions A.*—(1) How do you propose to carry out the experiment?

(2) Must the cork that carries the candle fit the lamp-glass quite air-tight?

(3) If you think you know, say why potash and calcium chloride are used.

5. Object.—To see whether the following bodies give off oxygen when heated in test-tubes: Mercuric oxide, potassium nitrate, red lead, barium peroxide, copper oxide, calcium oxide (lime), potassium permanganate, manganese dioxide, potassium chlorate.

All these bodies *contain* oxygen.

Describe fully all the changes observed, and pay particular attention to the ease with which the gas is given off.

6. Object.—To study the action of heat on mercuric oxide.

The information you are asked to obtain by experiment is, what volume of gas is given off, and how much the mercuric oxide loses in weight. (A suitable quantity of mercuric oxide to take is 2 gms.)

*Questions A.*—(1) Draw the simplest form of apparatus you would use, and say how you would use it?

(2) If the method, though simple, is not very accurate, point out where the fault lies.

7. The preparation of oxygen.

*Preparation.*—Mix 10 parts potassium chlorate with 3 parts of manganese dioxide. Grind in a mortar, and place in the test-tube A (see diagram). Then heat gently. Weigh the manganese dioxide carefully before mixing. Weigh again after dissolving the potassium chloride from it.

*Question A.*—Will you collect *all* the gas that comes off?

Burn carbon, sulphur, and phosphorus in the oxygen. The substances to be burnt are placed in a deflagrating spoon.

(*a*) After burning carbon add lime-water.

(*b*) Repeat with another jar, but add litmus solution.

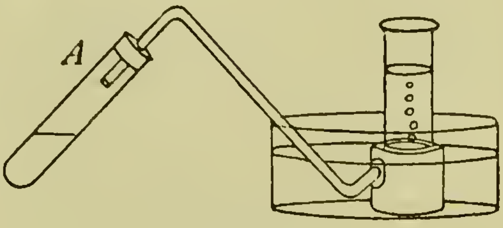

Fig. 4.

(*c*) After burning sulphur, add water and shake up, keeping the hand tight over the mouth of the jar. Then add litmus solution.

(*d*) Repeat (*c*) with phosphorus in place of sulphur.

8. Object.—(*a*) To find the weight of oxygen driven off from potassium chlorate by heat.

*Questions A.*—(1) How do you propose to do this simple experiment?

(2) What precautions must you take?

(*b*) To find the volume of oxygen given off.

*Question A.*—Look at the figure of the apparatus, then say how it can be used to find the volume of gas given off.

(*c*) Dissolve the residue in water and add silver nitrate solution.

Do the same with some potassium chlorate and compare.

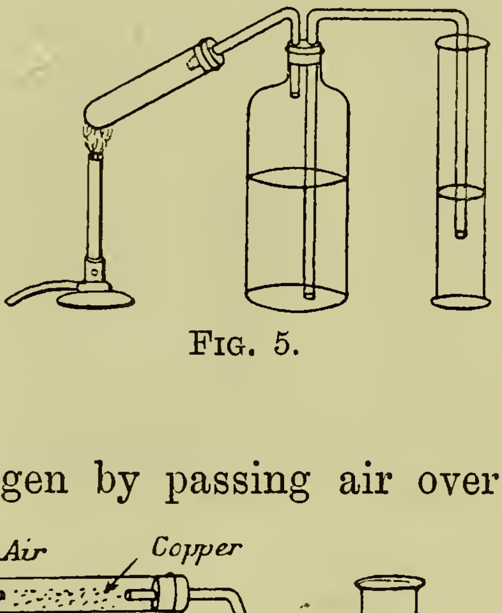

Fig. 5.

9. Object.—To prepare nitrogen by passing air over copper.

The air may be driven out of a bottle by running in water from the tap. Fit up the apparatus shown. Try burning a splint in the gas. Add lime-water.

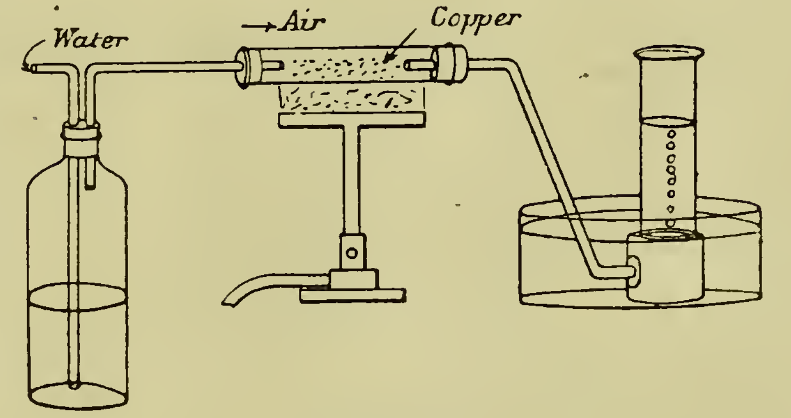

Fig. 6.

*Question B.*—What gases have you proved that it is not?

**10. Object.**—To measure the volume of nitrogen in air.

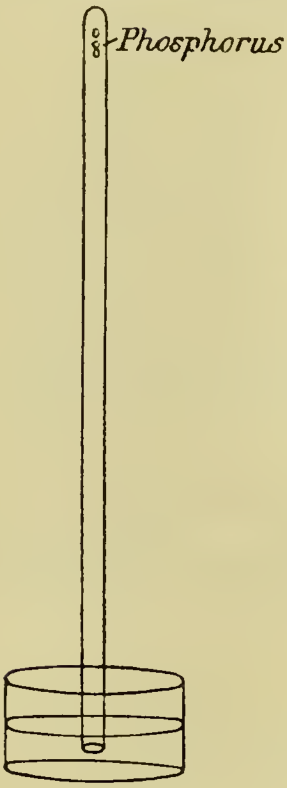

Fig. 7.

This can be done very easily by allowing phosphorus to smoulder in the air until the oxygen has all been removed. Fill a long tube containing a lump of phosphorus at its closed end, with water, pour out the water so that the tube fills with pure air, and immediately put the open end under water. Leave the apparatus for half an hour, then describe exactly the changes which have taken place. See if the water has changed in any way.

*Question B.*—What is the advantage of the narrow tube?

**11. Object.**—To study the formation of rust.

(*a*) To see whether air is used up when iron rusts. Use iron filings (why?). Place them in a closed volume of air and see if it diminishes at all in the course of a week.

*Question A.*—Devise your own apparatus.

(*b*) You know that iron rusts in a damp place quicker than in a dry one. See if iron rusts in quite dry air.

(*c*) Find out if iron gains in weight when it rusts.

Leave some filings in a moist condition. Weigh them a week later and see if they have changed in weight.

What precautions must you observe?

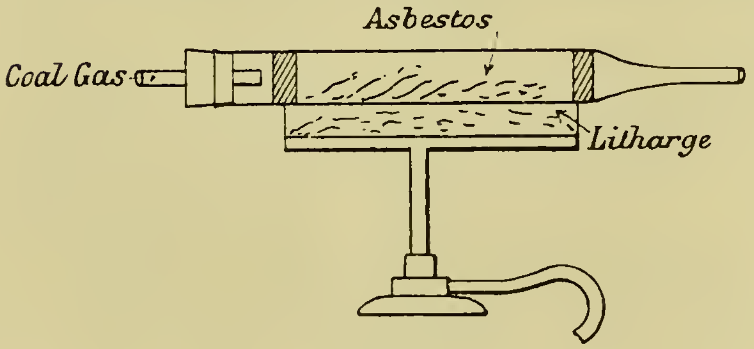

Fig. 8.

**12. Object.**—To obtain metals by reduction.

(*a*) Pass a current of coal gas over litharge (use a tube of the following shape).

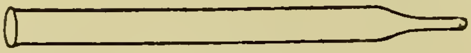

Fig. 9.

*Questions A.*—(1) What is the asbestos for?

(2) What is the actual change taking place?

(*b*) Heat up 3 gms. of litharge with 1 gm. of carbon and 2 gms. of fusion mixture as strongly as possible in a crucible.

*Question A.* (1)—What burner will you use?

(2) Having got your metal, how will you show that it is really lead?

## SECTION II.—Common Acids and Alkalies.

**13.** Object.—To examine the effect of heat upon ferrous sulphate crystals.

(*a*) Heat gently and notice any changes that take place.

(*b*) Heat more strongly and raise the temperature as high as possible.

*Question A.*—What apparatus will you choose for *a* and *b*?

Test any fumes given off, with litmus. Smell them.

See whether the substances remaining after (*a*) and (*b*) dissolve in water.

*Question B.*—What are the properties by means of which you recognise an acid?

**14.** Object.—Experiments with sulphuric acid.

(*a*) Add about 10 c.c. of strong sulphuric acid to a test-tube half full of water.

(*b*) Put a layer of cane-sugar in the bottom of a beaker and just cover it with sulphuric acid.

(*c*) Write your name on a spare piece of paper, using solution (*a*) instead of ink; warm the paper very slightly.

(*d*) Evaporate a few drops of solution (*a*) in a porcelain basin.

(*e*) Add a few drops of barium chloride solution to 1 c.c. of solution (*a*).

(*f*) Add a few drops of lead nitrate solution to 1 c.c. of solution (*a*).

**15.** Object.—To prepare hydrochloric acid.

(*a*) Add a few drops of strong sulphuric acid to salt (sodium chloride).

(*b*) Add a few drops of strong sulphuric acid to ammonium chloride.

(*c*) Add a few drops of strong sulphuric acid to calcium chloride.

Warm gently in each case.

*Question B.*—What happens?

(*d*) Set up the apparatus figured below.

Collect three jars of the gas. Find out yourself whether the gas dissolves in water, and whether it burns or supports combustion.

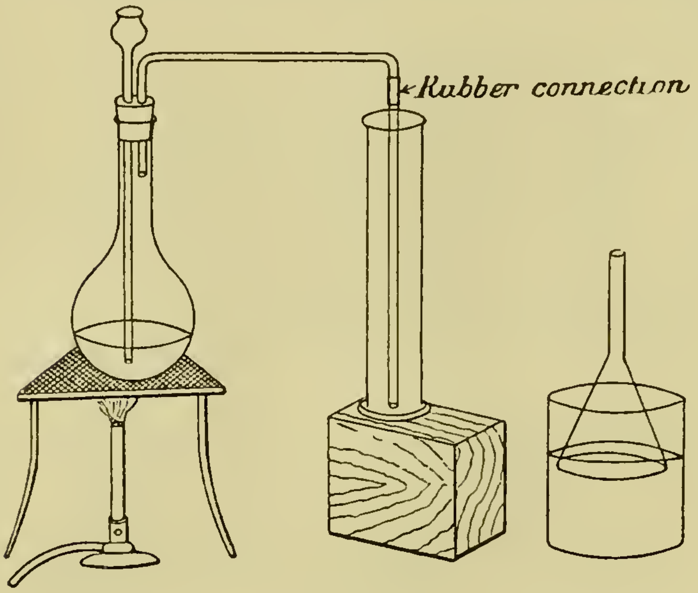

FIG. 10.

Pass the gas into water, using the beaker and funnel arrangement (see diagram).

Amounts to be used—

30. *gms. of sodium chloride; cover with strong sulphuric acid.*

Bring the stopper of an ammonia bottle near to one of the jars. Into an empty jar pour a few c.c. of strong ammonia, bring one of the jars of hydrochloric acid over it, quickly remove the plate, and allow the vapours to mix. Evaporate a little of the liquid in the beaker. To various parts of the liquid in the beaker add 1 c.c. of solutions of silver nitrate, barium chloride, and lead nitrate respectively.

*Questions B.*—(1) Why does the gas remain in the jar?

(2) Why is a funnel used when the gas is passed into water?

(3) How could you distinguish hydrochloric acid from sulphuric acid?

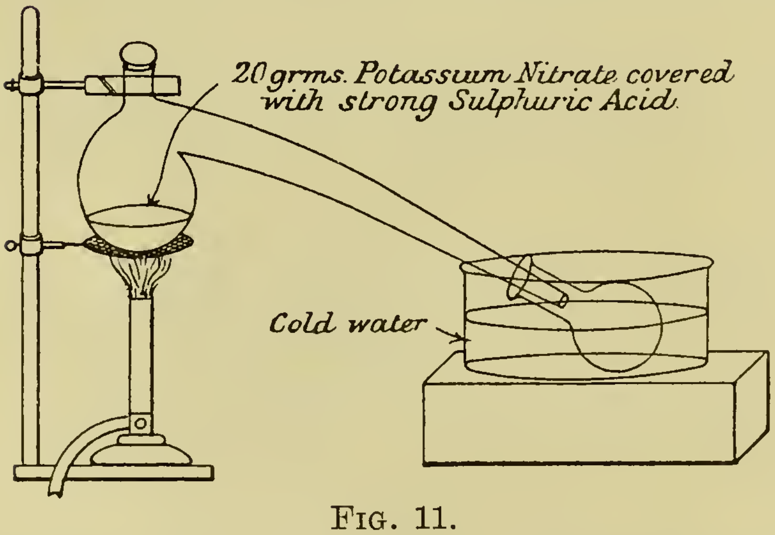
20 grms. Potassium Nitrate covered with strong Sulphuric Acid
Cold water

FIG. 11.

**16.** OBJECT.—To prepare nitric acid.

Add a few drops of strong sulphuric acid to (*a*) nitre (saltpetre, potassium nitrate), (*b*) chili saltpetre (sodium nitrate), (*c*) lead nitrate. Warm gently in each case and describe what happens. (*d*) Fit up a retort as shown. Warm gently at first, then more strongly, but do not let the fumes come off so rapidly that they escape from the mouth of the flask.

Test the distillate in the flask with litmus.

See what its action is on (*a*) paper, (*b*) a finger-nail, (*c*) copper, (*d*) sugar.

*Question B.*—What remains behind?

17. Object.—The preparation and properties of carbonic acid.

Add a few drops of strong hydrochloric acid to a little (*a*) calcium carbonate, (*b*) magnesium carbonate, (*c*) copper carbonate, (*d*) zinc carbonate.

*Question B.*—Is a gas given off; if so, does it put out a lighted splint?

Fit up the apparatus shown. Into the Woulff's bottle place small lumps of marble, cover them with water, then pour strong hydrochloric acid in small quantities at a time down the thistle funnel. Collect the gas that comes off by "downward displacement" (as in figure). Test to see whether jar is full by means of a lighted splint. With successive jars of the gas find out—

(1) The action of the gas on lime-water.

(2) The action of the gas upon litmus.

(3) Whether the gas dissolves at all in water. Do it by shaking up the jar of gas with water, keeping the mouth of the jar under water all the time.

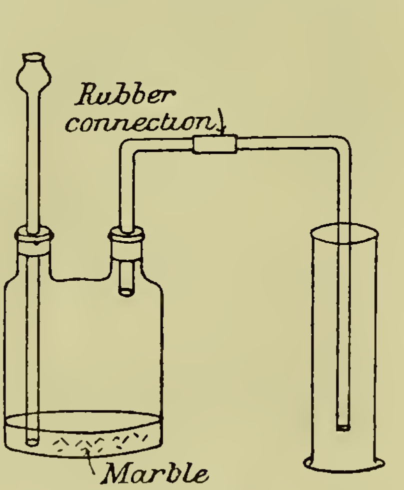

Fig. 12.

(4) Repeat (3), but use dilute caustic soda (1 per cent. solution).

(5) Say whether the gas is heavier or lighter than air. (Think of a very simple way of doing this.)

(6) Bubble the gas slowly for a long time through lime-water.

*Question B.*—(1) Do you consider the solution, in water, a strong acid?

(2) What is formed when the gas is passed into lime-water (1 and 6) and sodium hydrate?

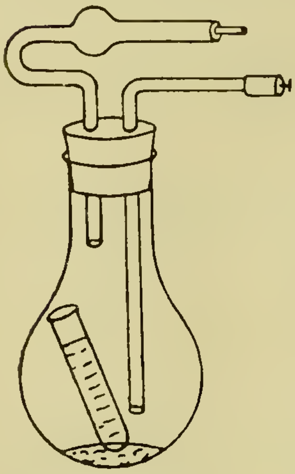

FIG. 13.

**17A.** OBJECT.—To find the weight of carbon dioxide given off when 100 gms. of calcium carbonate dissolve in hydrochloric acid.

Use the apparatus shown, and write out the method in detail before starting. The bottle or flask contains calcium carbonate and water. The tube inside contains strong hydrochloric acid. The tube on the top contains calcium chloride.

**17B.** OBJECT.—Find the loss in weight of 100 gms. calcium carbonate on strongly heating.

The method is obvious; write it out, nevertheless, before starting. You will want to examine the residue later, therefore cork it up immediately, in a specimen tube.

**17C.** OBJECT.—Find the volume of carbon dioxide evolved from 100 gms. of calcium carbonate. Use the method of water displacement.

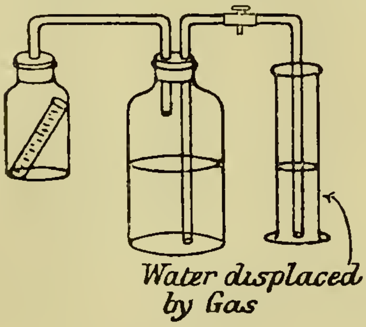

FIG. 14.

Place the marble and a little water in the small bottle, and strong hydrochloric in the little tube.

**18.** OBJECT.—To examine the action of sulphur on iron at a red heat.

Thoroughly mix the two in a test-tube, then heat strongly and watch. Break open the test-tube when it is cold, and describe the result.

.Place a few grains of the sulphide of iron you have made in a test-tube, add a little hydrochloric acid, describing fully all the changes you can observe, not omitting to notice if there is any peculiar odour.

Fit up another test-tube with a cork and delivery tube, and place in it some of the iron sulphide and hydrochloric acid. (Use the fume cupboard.) Pass the gas into (*a*) water, add litmus and notice carefully if it changes colour, (*b*) dilute copper sulphate solution, (*c*) cadmium chloride, (*d*) antimony chloride, (*e*) lead acetate, (*f*) dilute caustic soda. Replace the delivery tube by a jet and see whether the gas will burn.

19. Alkalies.—Add water drop by drop to the residue from 17B, noting any change that takes place very carefully. Then shake up with water and filter.

(*a*) To one part of the solution add a few drops of red litmus.

(*b*) Taste the solution.

(*c*) Blow through another part of the solution (air from the lungs contains carbonic acid).

20. Object.—To examine soda and potash.

Obtain some washing-soda and some potash.

*Question A.*—In what respects do they differ, *e.g.* appearance, taste, "feel," solubility in water, action of heat, action on litmus, action of hydrochloric acid ?

21. Object.—To prepare sodium hydrate.

Shake up a strong solution of sodium carbonate with lime, allow the sediment to settle, decant the clear solution. Feel it and examine its action on litmus. Evaporate to dryness in a porcelain basin and continue heating. Allow to cool and examine the residue. Dissolve out the residue by heating with water, and see whether the basin is roughened and corroded inside.

22. Object.—To prepare ammonia gas.

Add a few drops of caustic soda to a little ammonium chloride in a test-tube, warm, hold a piece of litmus paper over the mouth of the test-tube, also carefully smell vapours given off.

Mix together 5 parts of dry ammonium chloride with 10 parts of lime, and place them in a test-tube fitted up as shown. Collect a jar of the gas by upward displacement and see if it is soluble in water (as you did with carbonic acid).

See whether it burns or supports combustion. Repeat the experiment with a jar of

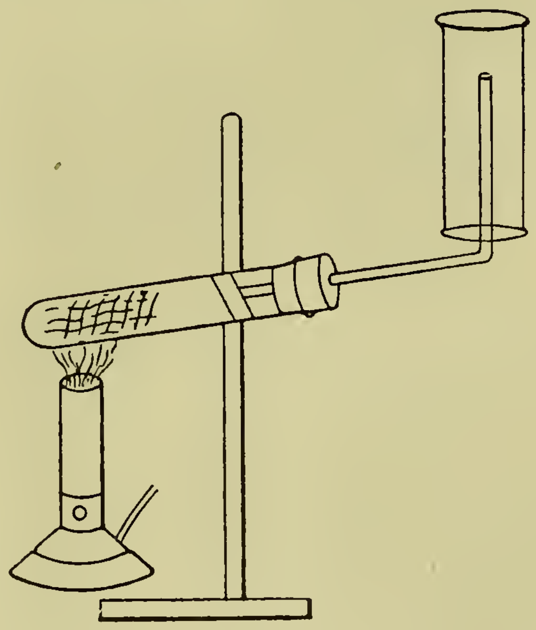

Fig. 15.

the gas and a jar of hydrochloric acid gas (obtained by putting a few c.c. of the strong acid at the bottom of a jar).

### SECTION III.—Salts.

**23.** Take 10 c.c. of a strong solution of sodium carbonate, add a few drops of litmus, and then hydrochloric acid until the solution *just* turns red. Evaporate to dryness and examine the residue.

**24.** Repeat, substituting sodium hydrate for carbonate.

**25.** Repeat, substituting ammonia for sodium carbonate.

**26.** Repeat, substituting lime for sodium carbonate. A strong solution cannot be made, therefore use the solid, dissolve in hydrochloric acid and evaporate to dryness. Dissolve a little of the dry substance, and add litmus to see if it is neutral.

Compare all these substances 23–26, appearance, taste, and action of heat.

**27.** Repeat 23, but use nitric acid instead of hydrochloric and potash instead of soda.

**28.** Repeat 24, but use nitric acid and caustic potash instead of caustic soda.

**29.** Repeat 25, but use nitric acid.

**30.** Repeat 26, but use nitric acid.

Compare as before.

**31.** Repeat 23, but take dilute sulphuric acid and neutralise still more carefully than in the previous cases.

**32.** Add lime-water to dilute sulphuric acid containing litmus, until neutral. Describe fully what happens.

**33.** **34.** **35.** While the solutions obtained above are evaporating, compare the strengths of the solutions given.

| | *A.* | *B.* |
|---|---|---|
| 33. | Sulphuric acid and | Sodium hydrate. |
| 34. | Hydrochloric acid and | Ammonia. |
| 35. | Nitric acid and | Potassium carbonate. |

The method is to take 20 c.c. of your alkali by means of a pipette, place it in a beaker with a little litmus and run the acid in very carefully from a burette until the colour of the litmus *just* changes to red. Express the result by saying that 1 c.c. of $A$ is neutralised by $x$ c.c. of $B$ ($x$ being the number of c.c. of alkali you find it necessary to run in $\div$ 20).

In each case evaporate the solutions to dryness on the water-bath, and obtain the weight of the solid.

### SECTION IV.—The Action of Dilute Acids on Metals.

As typical metals, take zinc, iron, magnesium, lead, copper, and tin. Place a little of each metal in a test-tube. Take cold acid first, then warm. Tabulate your results, showing which metals dissolve, which give off gases, whether the gas supports combustion or burns, or has any other distinctive features, such as colour or smell.

36. Sulphuric acid.

37. Hydrochloric acid.

38. Nitric acid.

39. Object.—The preparation of hydrogen.

Place zinc in the Woulff's bottle (see figure) to depth of about half an inch. Cover the zinc with water, then pour strong sulphuric acid down the funnel, not more than 1 or 2 c.c. at a time.

Collect 4 jars of the gas, discard all those that explode or whistle on applying a light. Show the lightness of the gas by leaving one jar mouth upwards and open. After a minute, apply a light. Do the same except that a jar is placed over the mouth of the jar of hydrogen and left so for two minutes. Collect one jar by upward displacement. Collect a dry test-tube full by upward displacement and burn it. Notice the dew formed.

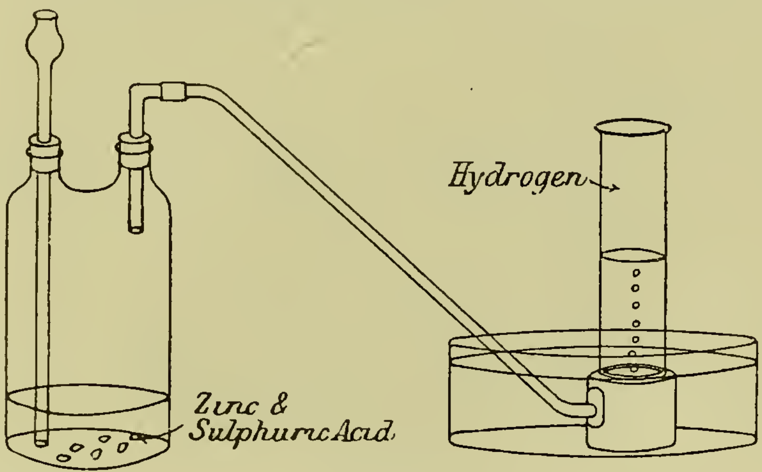

Fig. 16.

Burn a jet of the gas—notice the moisture it deposits on a cold surface. Hold a long wide glass tube over it and try to obtain a "singing flame." Filter the solution left in the bottle and crystallise it.

40. Object.—To find the weight of oxygen that combines with 1 gm. of hydrogen.

Fit up apparatus as shown in the diagram. Place a weighed quantity of copper oxide [granular] in the porcelain boat *d*. Weigh the bulb *e* and tube *g*, and then pass a slow current of pure dry hydrogen over the heated copper oxide. When the oxide has been reduced to copper, turn out the flame and allow the tube *c* to cool down. Displace the hydrogen by drawing a slow stream of air through the apparatus by means of an aspirator. Then reweigh the porcelain boat and its contents and the bulb *e* and the tube *g*. The loss in weight of *d* will be the amount of oxygen gone to form water, and the gain in weight of *e* and *g* is the weight of water produced. From your results deduce the chemical composition of water, giving your answer in percentages.

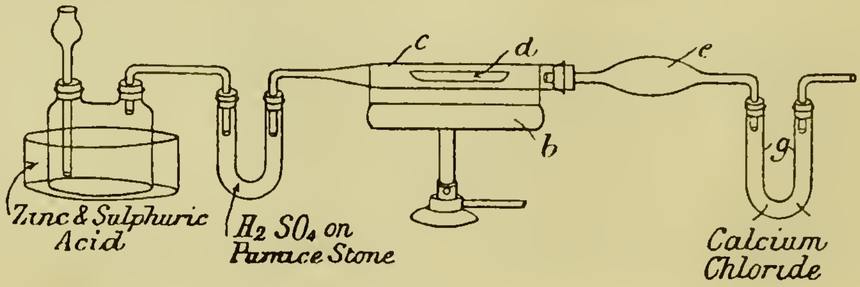

FIG. 17.

*Question A.*—How will you know when the oxide is all reduced?

*Questions B.*—(1) Why are you asked to pass a slow current of hydrogen over the heated copper oxide?

(2) Why is a stream of air drawn through the apparatus before the final weighings?

(3) Why is a stream of hydrogen passed through the tube *c* while it is cooling down?

(4) What sources of error do you think arise in this experiment?

41. To collect the products formed when hydrogen is burnt. The hydrogen is generated by a Kipps apparatus,

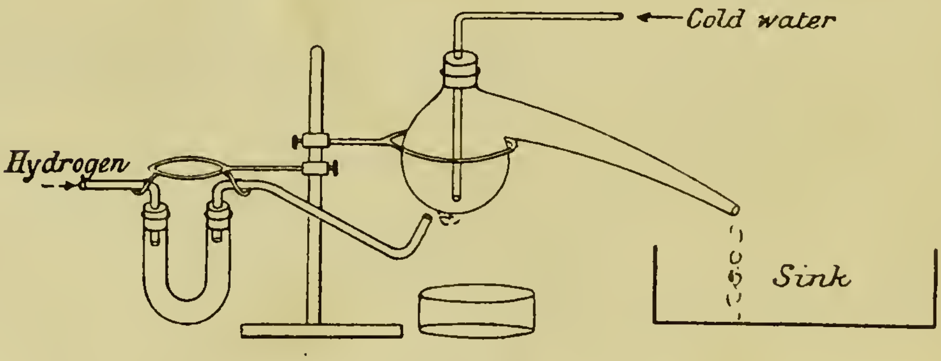

FIG. 18.

passed through a drying tube, and burnt at a jet as shown in the figure, so that the products of combustion are condensed on the cold retort and collected in a dish.

Notice the taste, smell, and colour of the liquid ; add a little of it to anhydrous copper sulphate. *If time permits*, weigh 1 c.c. of it and also determine its boiling-point. If convenient, do a melting-point determination as well.

42. Object.—Decompose water by sodium and calcium.

Obtain a fairly large dish of water [about 6 ins. diameter], and float bits of sodium and calcium on the water. Put a lighted match near them as they float about. Place a piece of sodium in a small piece of lead piping closed at one end. Place in dish, and hold a test-tube full of water over the end so as to collect the gas evolved. Test the gas to see if it is hydrogen.

*Question B.*—Is there any difference between the action of the *two metals* on water ?

43. Object.—Prepare hydrogen by passing steam over red-hot iron.

Fit up the apparatus shown in the diagram. Heat the iron tacks in *B* until they are at a red heat, and then pass steam generated in the flask *A* over the heated iron. Collect the gas in the jar *C*. Neglect the first jar collected. Test the gas to show that it is hydrogen, and in such a manner as to distinguish it from other gases which resemble it.

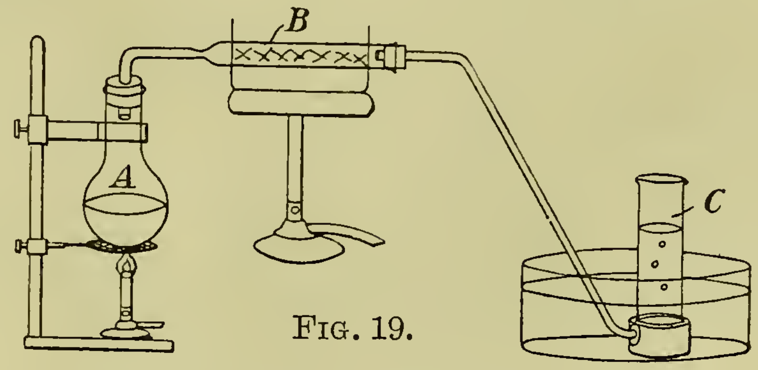

Fig. 19.

*Questions B.*—(1) Did you test your apparatus to see if it was air-tight before commencing the experiment ? If so, how ?

(2) Why are you asked to neglect the first jar full of the gas collected ?

(3) What tests did you do with the gas to show it was hydrogen ?

## SECTION V.—The Action of Strong Acids on Metals.

Repeat the Exps. 36, 37, 38, but now use strong acids.

**44.** Using hydrochloric acid.

**45.** Using sulphuric acid.

**46.** Prepare sulphur dioxide.

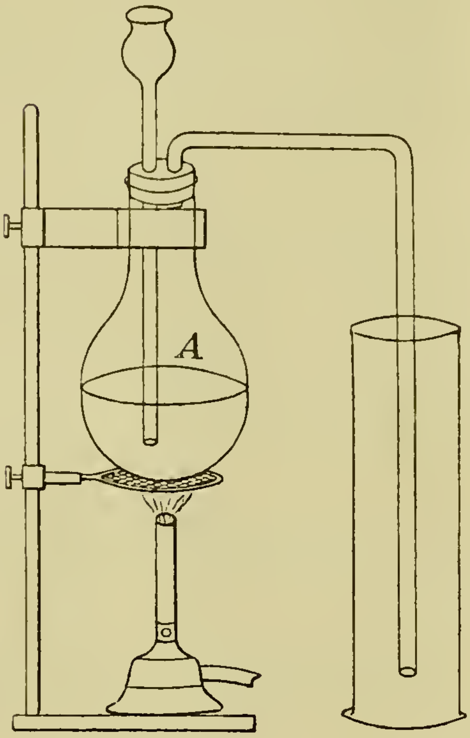

Fig. 20.

Fit up the apparatus shown in the diagram. The flask *A* contains copper turnings. Strong sulphuric acid is poured down the thistle-headed funnel and the mixture warmed. Sulphur dioxide is produced. Notice smell.

Test with a lighted taper to see whether it supports combustion or burns.

See whether it will dissolve in water [same method as for $CO_2$]. Notice the action on litmus.

Obtain some flowers, and see whether it will bleach them.

Add a weak solution of potassium permanganate to a solution of the gas in water. Test with barium chloride before and after adding the potassium permanganate.

**47.** Using nitric acid (as in 44).

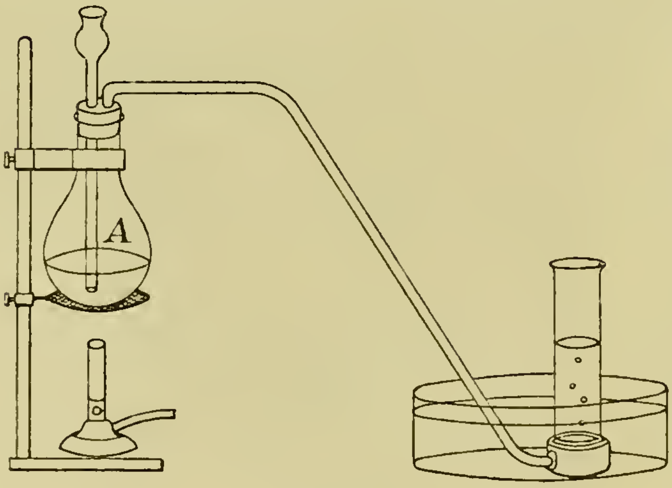

Fig. 21.

**48.** Preparation of nitric oxide.

Fit up the apparatus shown in diagram. Place copper turnings in flask *A* and cover with a layer of water. Then pour strong nitric acid down the funnel and collect the gas evolved over water. Collect 4 jars.

Open one to the air.

Allow a little air to enter a jar half full of the gas as it stands in the pneumatic trough.

Allow a little oxygen to enter a jar half full of the gas as it stands in the pneumatic trough.

Add neutral litmus solution to the water that has dissolved the brown fumes.

*Problems.*

**49.** OBJECT.—Investigate the action of dilute nitric acid [1 : 10] on zinc.

**50.** Preparation of nitrous oxide.

Heat a little ammonium nitrate in a test-tube and examine the gases given off, particularly with regard to power of supporting combustion.

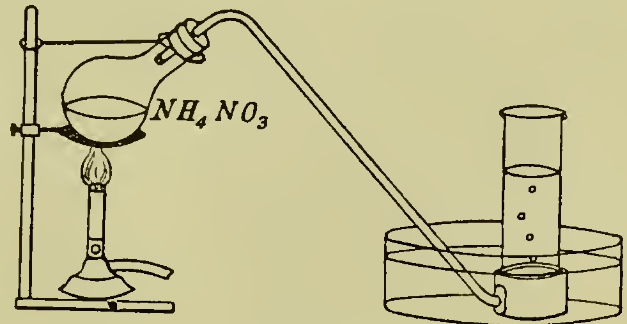

FIG. 22.

Prepare the gas on a large scale, using the apparatus shown in the diagram. Use *hot* water in the pneumatic trough. Collect four jars.

Try introducing a glowing splint into the jar.

See whether carbon, sulphur, phosphorus will burn in it.

Form some idea as to its solubility in cold water.

## SECTION VI.—Equivalent Weights.

### *Explanatory Notes and Examples.*

The following is the general definition :—" The equivalent weight of an element is that weight of the element which will combine with or replace one gram of hydrogen."

In the following experiments four different methods are employed to find the equivalent weights of elements :—

(*a*) By collecting the gas or the water displaced by the gas when hydrogen is turned out by means of a metal.

(*b*) By causing a metal to unite with oxygen [assuming the equivalent weight of oxygen].

(*c*) By removing oxygen from an oxide and leaving the metal.

(*d*) By replacement from solution of one metal by another.

1 litre of hydrogen weighs 0·0896 gm.
1 gm. occupies 11·2 litres.

*Examples.*—(*a*) Suppose 0·5 gm. of magnesium be taken and 465 c.c. of hydrogen be generated.

Now, 465 c.c. of hydrogen weigh $0{\cdot}09 \times \frac{465}{1000} = 0{\cdot}041$ gm.

Then, since 0·041 gm. of hydrogen is set free by 0·5 gm. of magnesium, 1 gm. of hydrogen is set free by $\frac{0{\cdot}5}{0{\cdot}041} = 12{\cdot}2$ gms. of magnesium.

∴ 12·2 *is the equivalent weight of magnesium.*

(*b*) For this method you are told that 8 is the equivalent of oxygen. The calculation therefore simply resolves itself into finding the number of gms. of magnesium which will unite with 8 parts of oxygen.

(*c*) In this method, suppose the following results have been obtained as the result of an experiment :—

| | | | | |
|---|---|---|---|---|
| (1) | Weight of copper oxide and boat | = | 20·00 | gms. |
| | Weight of boat | = | 18·42 | ,, |
| | Weight of copper oxide taken | = | 1·56 | ,, |
| (2) | Weight of boat and copper | = | 19·68 | ,, |
| | Weight of boat | = | 18·42 | ,, |
| | Weight of copper in 1·58 gm. of copper oxide | = | 1·26 | ,, |
| | Weight of oxygen ,, ,, ,, | = | 0·32 | ,, |

Again, assuming 8 as the equivalent weight of oxygen, all that is required is to calculate how many parts of copper unite with 8 parts of oxygen :—

$$\frac{8 \times 1{\cdot}24}{0{\cdot}32} = \underline{31{\cdot}5}$$

(*d*) For this method the equivalent of one metal is given, the calculation therefore resolves itself into calculating how many parts of the other metals are equivalent to the number given, *e.g.* :—

Ag = 108 . 0·5 gms. of copper turned out 1·70 gms. Ag. Find the equivalent weight of copper.

$$\text{Equivalent weight of copper} = \frac{108 \times 0{\cdot}5}{1{\cdot}70} = \underline{31{\cdot}5.}$$

**51.** Object.—Find the equivalent weight of magnesium by collecting the hydrogen evolved when it dissolves in hydrochloric acid.

*Apparatus.*—Burette, 2 ins. narrow rubber tubing, glass funnel, clip, porcelain dish, retort stand, and ring. Arrange as shown in the diagram.

*Questions A.*—(1) How much magnesium do you propose taking?

(2) Will you use strong or weak HCl to dissolve the metal?

*Method.*—Nearly fill the porcelain basin with water, and then remove the funnel and suck water up the burette until it reaches the last graduation. Keep in that portion by means of the clip. Then insert the weighed quantity [you are not required to weigh the magnesium yourself] of magnesium supplied to you as shown in the diagram. Now replace your funnel and partly fill with the HCl. Open clip and allow acid to run in the burette. Measure the volume of hydrogen produced, and from this calculate the equivalent weight of magnesium. [See definition.]

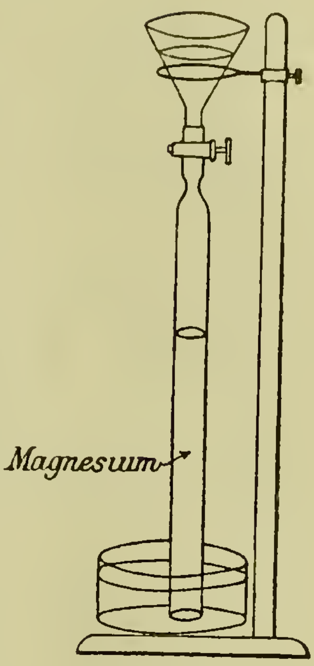

FIG. 23.

*Questions B.*—(1) Can you suggest any probable sources of error in this experiment? If so, how would you correct for same?

(2) Why are you not required to weigh your own magnesium?

52. Find the equivalent weight of magnesium by igniting it in air.

*Apparatus.*—Crucible and lid, pipeclay triangle, tripod, tongs.

*Questions A.*—(1) What weight of magnesium do you propose to take?

(2) What does the magnesium form when it is heated in contact with the air?

(3) What precautions must you take during the heating of the magnesium?

*Method.*—Weigh the crucible alone, and then again containing the magnesium turnings supplied to you. Ignite cautiously in contact with the air. The action is complete when the metal no longer glows brightly. Find the weight of the compound produced, and from this calculate the equivalent of magnesium.

*Questions B.*—(1) Why are you asked to weigh the magnesium in a crucible?

(2) What sources of error are likely to arise in this experiment?

**53.** Object.—Find the equivalent weight of zinc by collecting the water displaced by the hydrogen produced when the metal is dissolved in an acid.

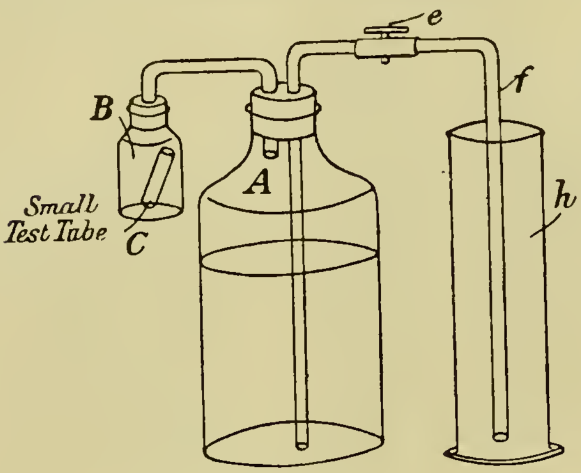

Fig. 24.

*Apparatus.*—Obtain the apparatus as seen in the diagram.

*Questions A.*—(1) It is necessary for the apparatus to be air-tight. How could you find out if this is the case?

(2) How much zinc would you propose to take, and why?

(3) Would you use strong or dilute acid? Give a reason for your answer.

*Method.*—After testing apparatus to see if it is air-tight, open the clip *e* and suck water into the tube *f* until it is full, then close clip. Fill the test-tube *C* with your acid and place in bottle *B* along with your weighed quantity of zinc. Replace cork in *B*, and then upset the acid in *C*, at the same moment opening the clip at *e*. Note the volume of water evolved. This is equal to the volume of hydrogen produced in the reaction. From this volume calculate an equivalent weight for zinc.

*Questions B.*—(1) Why is it necessary to fill the tube *f* with water before commencing the experiment?

(2) What sources of error may arise during this experiment, and how could they be removed?

**54.** Find the equivalent weight of aluminium by collecting the hydrogen evolved when the metal dissolves in sodium hydrate.

*Question A.*—Could the equivalent weight of copper be determined in the same way?

*Apparatus.*—Hard glass tube and delivery tube as shown in diagram at *A*. Beaker containing sodium hydrate, tripod, and wire gauze, pneumatic trough, beehive shelf, graduated glass cylinder. Arrange as shown in the diagram.

Take a weighed quantity [not more than 0·2 gm.] of aluminium and place in the hard glass tube, taking care to replace the copper plug. Nearly fill the beaker *B* with strong solution of sodium hydrate. Fill your glass cylinder with water, and place the end of the delivery tube under the mouth of the cylinder on TOP of the beehive shelf. Then gently warm the sodium hydrate, taking care not to boil, and collect the hydrogen evolved. From this volume calculate the equivalent weight of aluminium.

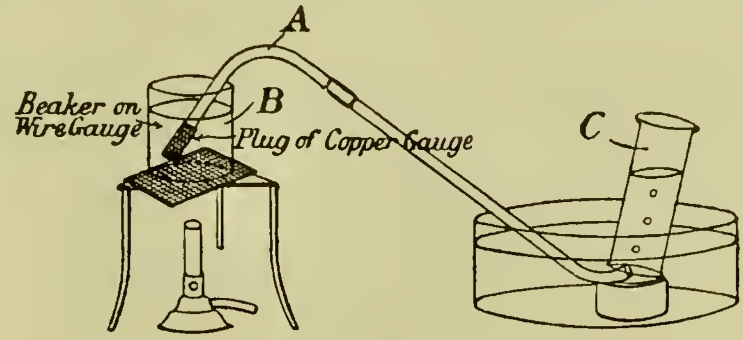

FIG. 25.

*Questions B.*—(1) Why are you asked to nearly fill the beaker *B* with sodium hydrate, and also to place the delivery tube on top of the beehive shelf?

(2) Why is a plug of copper gauze used?

(3) What sources of error are likely to arise during the experiment?

55. OBJECT.—Find the equivalent weight of copper by reduction of its oxide with hydrogen.

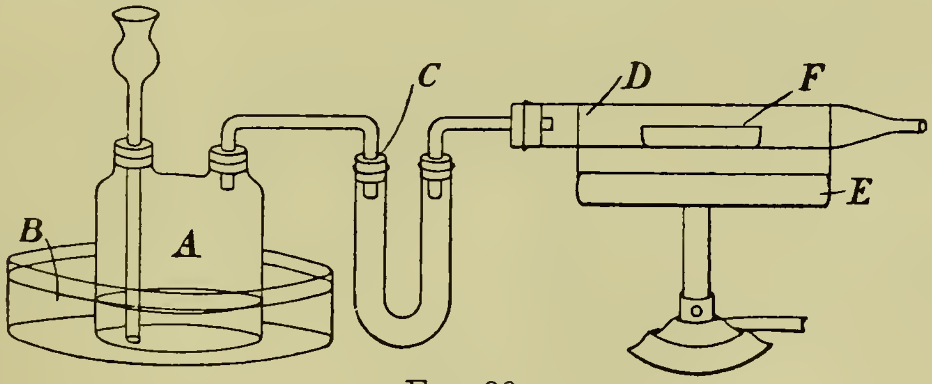

FIG. 26.

*Apparatus.*—Hydrogen generator *A*, dash of water *B*, tube containing pumice stone moistened with strong $H_2SO_4$ *C*, hard glass tube *D*, Ramsay burner *E*, porcelain boat *F*. Arrange as shown in the diagram.

*Questions A.*—(1) Why are you asked to stand the hydrogen generator in a dish of water?

(2) What use is the tube *C*?

(3) Is it better to take a large or small quantity of copper oxide?

*Method.*—Weigh your copper oxide in the boat *F*, and then place in the hard glass tube *D*. Now pass hydrogen over the oxide. Before lighting the burner *C* test your hydrogen to see if it is perfectly pure. After all the oxide has been reduced turn off the burner *E*, but do not stop your current of hydrogen until the tube *D* is quite cold. From the weight of copper obtained calculate the equivalent weight of that metal.

*Questions B.*—(1) How did you test your hydrogen to see if it was pure?

(2) Did you notice any striking change during the passing of the hydrogen over the heated oxide?

(3) Why haven't you to stop the hydrogen until the tube *D* is quite cold?

(4) Why is it necessary to have dry hydrogen for the reduction?

**56.** Find the equivalent weight of copper by replacement.

*Apparatus.*—Basin containing silver nitrate solution and pieces of copper foil.

*Questions A.*—(1) Would you take a small or large quantity of copper? Give reason for your answer.

(2) Is it better to use a strong or a dilute solution of silver nitrate?

*Method.*—Place a weighed quantity of copper foil in the solution of silver nitrate, and allow it to completely dissolve. [If the copper will not dissolve, you must decant the liquid off, taking care not to lose any silver, and add a fresh quantity of solution.] When all copper has dissolved, decant the liquid and wash twice with water by decantation. Dry very carefully over the Bunsen burner. From the weight of silver obtained calculate the equivalent weight of copper, assuming that of silver is 108.

*Questions B.*—(1) Did you notice any change in the appearance of the solution? If so, what reason would you assign for same?

(2) What chemical change has taken place during the experiment?

**57.** Find the equivalent weight of zinc by replacement.

*Apparatus.*—Basin containing copper sulphate solution, zinc foil.

*Questions A.*—(1) Would you take a small or large quantity of zinc?

(2) Is it better to use a strong or dilute solution of copper sulphate?

*Method.*—Exactly the same as in the previous experiment, except that the residue of copper, after being washed with water, should be washed once with alcohol and then dried very carefully. Assuming the equivalent weight of copper to be 31·5, find the equivalent weight of zinc.

*Questions B.*—(1) Why must great care be taken when drying the residue of copper?

(2) Did you notice any change in the appearance of the solution? If so, what reason could you assign for same?

(3) Could you suggest any reason for washing the residue of copper with alcohol?

(4) What chemical change has taken place during the experiment?

## SECTION VII.—Experiments on the Halogens.

**58.** Object.—Preparation of Hydrochloric Acid. [See Exp. 15.]

**59.** Object.—To notice action of oxidising agents on hydrochloric acid.

*Method.*—Take about 1 c.c. of dilute hydrochloric acid and warm with the following compounds containing oxygen, potassium nitrate, mercuric oxide, red lead, potassium chlorate, barium peroxide, potassium permanganate, potassium bichromate. Notice in particular the action on red litmus, and the smell.

**60.** Object.—Preparation of chlorine.

*Method.*—Fit up apparatus shown in diagram, and place granular manganese dioxide in flask *A*. Moisten with water, and then pour strong hydrochloric acid down the thistle funnel. Warm, and collect chlorine evolved by downward displacement. Collect three jars.

Notice colour, smell, etc.

Put powdered antimony in one jar. Note what happens.

Try action on litmus paper dry and wet.

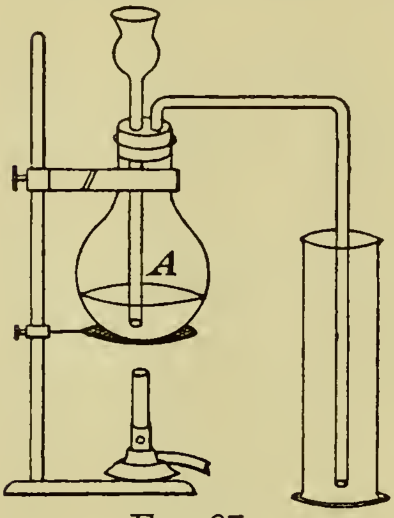

Fig. 27.

Write on a piece of printed paper, moisten and place in a jar of the gas. Note what happens.

See if a burning splinter will continue to burn in the gas.

*Preparation of chlorine water.*—Pass the gas into water and obtain a strong solution. Notice smell, colour, action on litmus.

Add a little to a solution of potassium bromide; add a little to a solution of potassium iodide. Note what happens in each case.

*Question.*—Can you explain the action of chlorine water on the above solution?

61. Object.—Examine the action of sulphuric acid on potassium bromide.

*Method.*—Place a little of the bromide in a test-tube; add sulphuric acid that has been diluted with an equal volume of water; warm. Notice the action of the fumes on blue litmus; notice the smell.

62a. Object.—Action of oxidising agents on hydrobromic acid.

*Method.*—Drop in a little manganese dioxide; record any change in the action on litmus, and in the appearance and smell. Then try the action of the strong sulphuric acid, undiluted, on potassium bromide, and compare.

Add a little chlorine water to potassium bromide.

62b. Object.—Action of sulphuric acid and oxidising agents on potassium iodide.

*Method.*—Do exactly the same as in 61, substituting potassium iodide for potassium bromide, but use (*a*) ordinary dilute sulphuric acid, (*b*) strong sulphuric acid.

62c. Object.—Action of oxidising agents on hydriodic acid.

*Method.*—Add bromine water to a solution of potassium iodide; notice any changes. Then add a few drops of a solution of starch in water.

## SECTION VIII.—Volumetric Analysis.

The equivalent weight of an acid is that weight in grams which contains 1 gm. of hydrogen replaceable by a metal.

(*a*) For example, hydrochloric acid ($HCl$) contains 1 gm. of replaceable hydrogen in 36·5 gms., therefore 36·5 is the equivalent weight of hydrochloric acid. Now, when an acid and a base neutralise each other, they always do so in the same definite proportions by weight.

(*b*) Thus 40 gms. of sodium hydrate always neutralise 36·5 gms. of hydrochloric acid.

$$\underbrace{NaOH}_{40} + \underbrace{HCl}_{36\cdot5} = NaCl + H_2O$$

Since 36·5 is the equivalent weight of hydrochloric acid [see above], 40 *is the equivalent weight of sodium hydrate.*

(*c*) Now, 53 gms. of sodium carbonate [$Na_3CO_3$] neutralise the same amount of hydrochloric acid.

$$\begin{array}{llll} Na_2CO_3 + & 2HCl = & 2NaCl + H_2O + CO_2 \\ \;\;106 & \;\;73 & \\ \;\;\;53 & \;\;36\cdot5 & \end{array}$$

∴ 53 *is the equivalent weight of sodium carbonate.*

*Example* (*a*).—Again, 40 gms. of sodium hydrate neutralise 49 gms. of sulphuric acid [$H_2SO_4$].

$$\begin{array}{lll} 2NaOH + & H_2SO_4 = & Na_2SO_4 + 2H_2O \\ \;\;\;80 & \;\;98 & \\ \;\;\;40 & \;\;49 & \end{array}$$

∴ 49 *is the equivalent weight of sulphuric acid.*

In the cases (*c*) and (*d*) the molecular weight is divided by 2 to obtain the equivalent weight, because in sodium carbonate there are two atoms of sodium replacing two of hydrogen, and in the sulphuric acid there are *two* replaceable *H* atoms.

In volumetric analysis solutions known as "normal" solutions are used. The definition of a normal solution is as follows:—

A "*normal solution*" is a solution which contains the equivalent weight of a substance in grams per litre.

Thus a normal solution of $H_2SO_4$ would contain 49 gms. per litre, *i.e.* 1 c.c. = 0·049 gm. of $H_2SO_4$;

One of sodium hydrate, 40 gms. per litre, 1 c.c. = 0·04 gm. $NaOH$;

One of sodium carbonate, 53 gms. per litre, 1 c.c. = 0·053 gm. $Na_2CO_3$.

A *decinormal* solution means $\frac{1}{10}$ normal.

A *centinormal* solution means $\frac{1}{100}$ normal.

The symbols used to represent the above are—

$$\text{Normal } N, \textit{ e.g. } N\ H_2SO_4.$$
$$\text{Decinormal } \frac{N}{10}, \textit{ e.g. } \frac{N}{10}\ NaOH.$$
$$\text{Centinormal } \frac{N}{100}, \textit{ e.g. } \frac{N}{100}\ Na_2CO_3.$$

Besides normal solutions, a solution simply of some arbitrary strength is often used. It is then called a standard solution. In this case, before proceeding with the experiment, find by calculation its strength in terms of the other substance used.

*Example.*—Find the strength of a given solution of sodium hydrate, using standard sulphuric acid, 1 c.c. = 0·0392 gm. $H_2SO_4$.

From equation, 49 gms. $H_2SO_4$ are equivalent to 40 gms. sodium hydrate.

$$\begin{array}{llll} H_2SO_4 & + 2NaOH & = Na_2SO_4 & + 2H_2O \\ 98 & 80 & & \\ 49 & 40 & & \end{array}$$

$$\therefore 0{\cdot}0392 \text{ gm. is equivalent to } \frac{40 \times 0{\cdot}0392}{49}$$
$$= 0{\cdot}032 \text{ gm. NaOH}$$

∴ 1 c.c. of standard $H_2SO_4$ is equivalent to 0·032 gm. of NaOH.

Suppose the result of the experiment was as follows:—

Mean of three titrations,* 20 c.c. of NaOH required 15·3 c.c. $H_2SO_4$.

$$\therefore \text{amt. of soda in 20 c.c. of solution} = 0{\cdot}032 \times 15{\cdot}3$$
$$\therefore \quad ,, \quad ,, \quad \text{1 c.c.} \quad ,, \quad = \frac{0{\cdot}032 \times 15{\cdot}3}{20}$$
$$= 0{\cdot}0245 \text{ gm. of NaOH}$$

*Questions.*—(1) What is the equivalent weight of potassium carbonate [$K_2CO_3$] and of potassium hydrate [KOH]? Show how you obtained your values.

---

* Titration denotes the finding the strength of one solution by means of another.

(2) Work out the following examples :—

(*a*) 20 c.c. of a solution of KOH are neutralised by 13·2 c.c. of a $N$ HCl solution. Find the number of grams of KOH per c.c. in the solution.

(*b*) 20 c.c. of a standard solution of sodium hydrate containing 0·035 gm. per c.c. are neutralised by 11·3 c.c. of a solution of $H_2SO_4$. Find the strength of the acid in grams per c.c.

Note.—(*a*) Always try and arrange your work so that your readings come between 10 c.c. and 25 c.c.

(*b*) Cleanliness of apparatus generally.

**63.** Object.—Study the behaviour of the indicators litmus, methyl orange, phenol phthalein, under various conditions.

*Apparatus.*—The following apparatus will be required for all the experiments in volumetric analysis : burette and stand, glass funnel, three flasks, pipettes.

(*a*) Note the colour of the three indicators supplied to you.

Then place about 5 c.c. of sodium hydrate in three test-tubes. To the first test-tube add two drops of litmus, to the second two drops of methyl orange, and to the third two drops of phenal phthalein. Note the colour produced in each case. Now add dilute hydrochloric acid to each tube until acid [slightly]. Again note the colours produced.

(*b*) *The case of strong acids and strong bases.*

Dilute the strong sulphuric acid at your bench three times [30 c.c. to 90 c.c.], and then titrate it against the ordinary solution of sodium hydrate supplied in the laboratory. Do this in the case of each indicator, and record your results.

(*c*) *The case of a weak acid and a strong base.*

Same as (*b*), given a carbonate of known strength and hydrochloric acid of known strength. Titrate, using each indicator, and compare each result with the correct value. In the case of litmus, try the effect of boiling throughout the titration.

N.B.—The weak acid is the carbonic, which remains in solution.

(*d*) *The case of a strong acid and a weak base.*

Same as in (*c*), using hydrochloric acid and the given ammonia solution.

(*e*) *The case of a weak acid and a weak base.*

Take 20 c.c. of the weak alkali used in the previous experiment (*d*), and titrate with the solution of oxalic acid provided in each of the three cases. From your results, draw conclusions with regard to the use of the indicators in a case such as this.

NOTE.—Take note that, when we say a *weak* or *strong* base, etc., the term does not imply a strong or dilute solution.

$H_2SO_4$ is a strong acid, whether the solution is dilute or otherwise.

NaOH is a strong base, whether the solution is dilute or otherwise.

$NH_4OH$ is a weak base, whether the solution is dilute or otherwise.

*Questions.*—(1) In the following cases which indicator would you use, and why?—

(*a*) Titrating a solution of $H_2SO_4$ against KOH.
(*b*) ,, ,, $K_2CO_3$ ,, HCl.
(*c*) ,, ,, oxalic acid against NaOH.
(*d*) Titrating a solution of oxalic acid against $NH_4OH$.

(2) Supposing you were given a solution of sodium carbonate, and you decided to find its strength by means of oxalic acid, which indicator would you use? Give reasons for your choice. Would you expect to get a good result?

**64.** Make a normal solution of sodium carbonate.

*Question A.*—What is a normal solution?

*Method.*—Weigh out 25 gms. of pure, dry sodium bicarbonate, and then heat in a porcelain dish until there is no further loss in weight. Always allow the dish and its contents to cool down in a desiccator. Now weigh out accurately 13·25 gms. of the sodium carbonate thus obtained on a watch-glass, and transfer it to a 250 c.c. measuring flask. Place a glass funnel in the neck of the flask, and gradually upset the solid into the funnel, washing it down into the flask with distilled water from a wash-bottle. [A spatula will be provided for knocking the solid from the watch-glass.] Every precaution must be taken not to lose ANY of the sodium carbonate. When all the sodium carbonate has been transferred into the flask, three parts fill the flask with distilled water and shake until all the carbonate is dissolved. Then make up to 250 c.c. and again shake, so as to get the solution thoroughly mixed. Now test this solution with the *N* HCl supplied, and see if it is normal. If it is found to be correct, place in a stoppered bottle, label, and put away to be used later.

*Questions B.*—(1) Why are you asked to weigh out 13·25 gms. of sodium carbonate?

(2) Write the equation representing the change which takes place when the bicarbonate is converted into carbonate.

(3) State what precaution you took when transferring the sodium carbonate from the watch-glass to the 250 c.c. flask.

**65.** Make a normal solution of sulphuric acid.

*Method.*—Measure out 7 c.c. of pure $H_2SO_4$, and pour slowly into about 150 c.c. of distilled water in a 250 c.c. flask. When the solution is quite cold, make up to the mark.

Now titrate this solution against your *N* $Na_2CO_3$ made in the previous experiment. You will find that the acid is too strong, and you must calculate how much water to add to bring it down to *N* strength.

*Example.*—Supposing it was found that 20 c.c. of *N* $Na_2CO_3$ were neutralised by 16·3 c.c. of the acid solution.

Now, if the acid were *N*, 20 c.c. of acid would neutralise 20 c.c. of $N_2CO_3$, ∴ it is evident that for every 16·3 c.c. of acid 3·7 c.c. of water must be added; ∴ the amount of water to be added $= \frac{(250 - 16{\cdot}3) \times 3{\cdot}7}{16{\cdot}3} = \frac{233{\cdot}7 \times 3{\cdot}7}{16{\cdot}3}$ $= 53{\cdot}06$ c.c.

Now add the calculated quantity of water, and do another titration to show that your acid is now normal. When correct, place in a stoppered bottle, label, and keep for use later.

*Questions.*—(1) Can you give any reason why 7 c.c. of acid are taken in the first case? The density of pure strong sulphuric acid is 1·84

(2) You are instructed to make your acid too strong. What reason can you give for being thus instructed?

(3) Which indicator did you use in the titrations?

**66.** Find the amount of sodium hydrate in grams per c.c. in a given solution, using the *N* acid made in Exp. 65.

*Method.*—The way in which this experiment is done is illustrated by the example on p. 42.

**67.** Find the amount of HCl in grams per c.c. in a given solution, using the *N* sodium carbonate made in Exp. 64.

*Method.*—Same as in Exp. 66.

**68.** Find the weight of potassium carbonate in the given tube, using standard $H_2SO_4$ [1 c.c. = 0·0061 gm. $H_2SO_4$].

*Method.*—Dissolve the $K_2CO_3$ in distilled water, taking care to wash out the tube. Then make up to 100 c.c. and titrate 20 c.c. at a time with the $H_2SO_4$ supplied. Then from your result calculate the amount of $K_2CO_3$ in the tube.

**69.** Find the percentage amount of pure HCl in the commercial acid.

N.B.—Commercial HCl contains ABOUT 30 per cent. of the pure acid, and its density is 1·14 (approx.).

*Method.*—Place some strong hydrochloric acid in a dry weighing-bottle of known weight, and weigh the bottle with its contents. Pour about 8 c.c. of the acid slowly into 90 c.c. of distilled water, reweigh the bottle with the residue of strong acid, and make the diluted acid up to 100 c.c.

N.B.—You must be very careful not to spill any of the strong acid; all that goes out of the weighed bottle must go into the water.

*Question.*—About how many grams of pure HCl are there in the 8 c.c. of concentrated acid?

Now titrate measured portions of the acid with standard NaOH, and calculate the proportion of real acid [HCl] with the acid of the following equation:—

$$NaOH + HCl = NaCl + H_2O$$

**70.** OBJECT.—Find the percentage amount of pure oxalic acid in the given crystals, using (*a*) litmus, (*b*) methyl orange, (*c*) phenol phthalein, and so test the indicators.

*Questions A.*—(1) What alkali do you propose to use in this experiment?

(2) Will you use strong or weak solutions of acid and base respectively?

*Method.*—Take a weighed quantity of the given oxalic acid and dissolve in water and make up to a known volume. Titrate measured portions of this solution against the standard alkali you have chosen, using each of the three indicators, and taking at least two readings in each case. Then calculate from your result the amount of oxalic acid in 1 gm. of the sample given for each of the three cases.

Oxalic acid $(COOH)_2$, sodium oxalate, $Na_2C_2O_4$, potassium oxalate, $K_2C_2O_4$.

*Questions B.*—(1) From your results, which indicator do you consider most suitable for this experiment?

(2) Do you think any of the three were entirely satisfactory?

71. Find the strength of the given STRONG ammonia solution in grams per 100 c.c.

*Method.*—Take 5 c.c. of the given ammonia solution, and, using the strongest standard acid supplied in the laboratory, do a rough titration to find the approximate strength of the ammonia solution. Then dilute it down so as to make it a convenient strength for doing your titrations. Calculate your result, using one of the following equations:—

$$2NH_3 + H_2SO_4 = (NH_4)_2SO_4$$
$$NH_3 + HCl = NH_4Cl$$

*Question.*—Do you think your result will be VERY accurate? If not, can you suggest any sources of error?

72. Find the strength of the given ammonium chloride solution [in grams per litre] by boiling out the ammonia with standard alkali.

*Method.*—Boil a measured volume of the given solution [about 20 c.c.] with a measured excess of standard solution of caustic soda [about 40 c.c.], until ammonia is no longer given off. If the residue is not strongly alkaline, add more alkali and reboil. Titrate the excess of fixed alkali * with standard acid, and work out your result, making use of the following equation:—

---

* At this stage it might be advisable to make up to a known volume, and titrate measured quantities with the acid.

$$NH_4Cl + NaOH = NaCl + NH_3 + H_2O$$

**73.** Find the strength of the given ammonium chloride solution [in grams per litre] by distilling the ammonia into standard acid.

*Apparatus.*—Fit up the apparatus shown in the diagram.

*Method.*—The ammonium chloride is boiled with soda, the ammonia driven off and absorbed by a measured excess of sulphuric acid, and the excess of sulphuric acid is then determined by titration. The acid that has been neutralised by the $NH_3$ can then be calculated, and the amount of ammonium chloride ascertained.

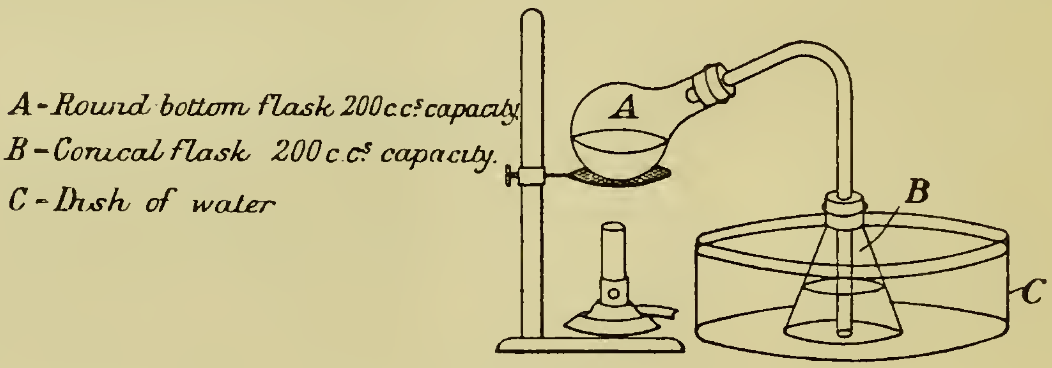
A - Round bottom flask 200 c.c.s capacity
B - Conical flask 200 c.c.s capacity.
C - Dish of water
A
B
C

FIG. 28.

*Method.*—Place 200 c.c. of the ammonium chloride solution along with excess of a solution of caustic soda [need not be of known strength] in the flask *A*. Then place a measured quantity [about 50 c.c.] of standard sulphuric acid in the flask *B*, which is standing in a dish of water *C*. Now heat the contents of *A* gradually to boiling, and keep at boiling-point for twenty minutes. Then stop heating, and at once remove the delivery-tube from out of *B*. The contents of *B* are then titrated with standard sodium hydrate, and thus the amount of acid not neutralised by ammonia ascertained. This taken from the amount first placed in *B* gives the amount neutralised by the ammonia gas. Then from the following equation:—

$$\begin{array}{ccc} H_2SO_4 + & 2NH_3 & = (NH_4)_2SO_4 \\ 98 & [2 \times 17] & \end{array}$$

the amount of ammonia produced can be calculated, and from this the quantity of $NH_4Cl$.

*Questions.*—(1) Which method of estimating the ammonium chloride do you think more accurate? Give reasons for your answer.

(2) How did you ascertain that ammonia was no longer given off in Exp. 72?

(3) What sources of error do you think might arise in the method adopted for Exp. 73?

(4) Why are you asked in Exp. 73 to remove the delivery tube from *B* at once?

**74.** Find the proportion of sodium carbonate in washing-soda, and deduce the formula of the salt.

*Method.*—Take some clean dry crystals of washing-soda, powder them, and weigh a convenient quantity, [about 30 gms.]. Dissolve this in water, and make up the solution to 200 c.c. Titrate 25 c.c. of this solution with your *N* acid [made in Exp. 65].

The method of calculation is as follows:—

Suppose that 20 c.c. of the solution containing 30 gms. of soda crystals in 100 c.c. are crystallised by 42·0 c.c. of $N\ H_2SO_4$. From the equation—

$$Na_2CO_3 + H_2SO_4 = Na_2SO_4 + CO_2 + H_2O$$

we see that 98 gms. of $H_2SO_4$ neutralise 106 gms. of $Na_2CO_3$.

But each c.c. of $N\ H_2SO_4$ contains $\frac{98}{1000 \times 2} = 0{\cdot}049$ gm. $H_2SO_4$ and will neutralise $\frac{106}{1000 \times 2} = 0{\cdot}053$ gm. $Na_2CO_3$

$\therefore$ 42 c.c. of $N\ H_2SO_4$ will neutralise $0{\cdot}053 \times 42 = 2{\cdot}226$ gms. of $Na_2CO_3$.

That is the amount of $Na_2CO_3$ in 20 c.c. of the original solution.

The amount in 100 c.c. is therefore 11·130 gms., and 30 gms. of washing-soda were used. The rest, therefore, must be water of crystallisation.

Then to find its formula, we have—

$$Na_2CO_3 = 11{\cdot}130 \div 106 = 0{\cdot}105$$
$$H_2O = 18{\cdot}870 \div 18 = 1{\cdot}05$$

But 0·105 : 1·05 : : 1 : 10

$\therefore$ formula of washing-soda is $Na_2CO_3 . 10H_2O$.

*Question B.*—(1) Why are you asked to take clean crystals of washing-soda?

(2) Could the formula of any other crystalline substance be deduced by the above method?

**75.** Find the basicity of the given acid whose molecular weight is 90.

*Question A.*—What is meant by the basicity of an acid? Give examples to illustrate your answer.

*Method.*—The solution supplied for this experiment contains half the molecular weight of the acid per litre; that is, 45 gms. per litre. Titrate measured quantities of this solution with standard sodium hydrate, and from your results calculate how many equivalents of soda are required to neutralise a molecular proportion of the acid. This will tell you the basicity of the acid.

*Question B.*—Could you determine the basicity of sulphuric acid, hydrochloric acid, and nitric acid by this method? Write down particulars in one case.

**76.** Find the percentage amount of barium chloride in 100 gms. of the given crystals—

(*a*) By precipitation with standard sodium carbonate.

(*b*) By dissolving the precipitate in standard acid.

*Question A.*—How do you propose weighing out the crystals?

*Method (a).*—Weigh out 2 gms. of the given crystals and dissolve in water, and then make up to 100 c.c. Then add to 20 c.c. of this solution 40 c.c. of a standard $Na_2CO_3$ solution. By this means the barium is precipitated as barium carbonate—

$$(a)\ BaCl_2 + Na_2CO_3 = BaCO_3 + 2NaCl$$

Filter off the precipitate, wash with water until the washings are free from alkali, then mix the washings and the original filtrate, and dilute the whole to a suitable volume. Find how much of the alkaline carbonate remains by titrating measured portions with standard acid. Deduct the excess of carbonate from the amount taken, to find how much was used by the barium chloride. Then from this, and using the equation, calculate the percentage of barium chloride in the given crystals.

*Method* (*b*).—Proceed as in the other method, only take the precipitate of barium carbonate, after washing, and dissolve in excess of standard acid. Then estimate the excess of standard acid with standard alkali solution. Deduct this from the amount of acid taken, and so obtain the amount of acid used in destroying the carbonate. Then, by means of the equation—

$$(b)\quad \underset{197}{BaCO_3} + \underset{73}{2HCl} = BaCl_2 + CO_2 + H_2O$$

calculate the amount of barium carbonate dissolved by the acid, and then from the equation given previously the amount of barium chloride in solution, and finally the percentage of barium chloride.

*Example.*—80 c.c. of standard $H_2SO_4$ [1 c.c. = 0·0392 gm.] were used to dissolve the precipitate [obtained from 50 c.c. of a solution containing 6 gms. of $BaCl_2$ per 100 c.c.], and the solution was then made up to 100 c.c. 20 c.c. of this solution required 10 c.c. of $N$ $Na_2CO_3$ to neutralise it.

Since 20 c.c. required 10 c.c. of $N$ $Na_2CO_3$ to neutralise it, 100 c.c. would require 50 c.c. of $N$ $Na_2CO_3$ to neutralise it.

Now, 1 c.c. of $N$ $Na_2CO_3$ is equivalent to 0·049 gm. $H_2SO_4$ [see p. 41]; therefore 50 c.c. of $N$ $Na_2CO_3$ is equivalent to 2·25 gms. $H_2SO_4$.

Amt. of $H_2SO_4$ used was 0·0392 × 50 = 3·136 gms.
,, ,, left [in excess] = 2·25 ,,
,, ,, used in dissolving $BaCO_3$ = 0·886 ,,

From equation (*b*), 73 gms. of HCl are equivalent to 197 gms. of $BaCO_3$.

$\therefore$ 0·886 gm. of HCl are equivalent to $\frac{197 \times 0·886}{73}$ gms. of $BaCO_3$ = 2·391 gms.

From equation (*a*), 197 gms. of $BaCO_3$ are produced by 210 gms. of $BaCl_2$.

$\therefore$ 2·391 gms. of $BaCO_3$ are produced by $\frac{210 \times 2·391}{197}$ of $BaCl_2$ = 2·549 gms. of $BaCl_2$.

The original solution was 6 gms. in 100 c.c., and 50 c.c. were used for the precipitation; that is, 3 gms. of $BaCl_2$

were taken. Amount found is 2·549 gms. The percentage of $BaCl_2$ in the given crystals is therefore—

$$\frac{2·549 \times 100}{3} = 84·9 \text{ per cent. approx.}$$

*Questions B.*—(1) Which method do you think is better for the estimation of $BaCl_2$ (*a*) or (*b*)? Give reasons in support of your claim.

(2) In method (*b*) how did you test the washings to see if they were free from alkali?

77. Find the amount of ammonium chloride in a given solution [in grams per litre], using $\frac{N}{10}AgNO_3$

(*a*) without the aid of an indicator.
(*b*) with " " "

*Preliminary Considerations.*—When silver nitrate solution is added to a solution of ammonium chloride, a white curdy precipitate of silver chloride is produced—

$$\underset{169·97}{AgNO_3} + \underset{53·5}{NH_4Cl} = \underset{80}{NH_4NO_3} + \underset{143·38}{AgCl}$$

By means of this reaction the amount of chlorine, and thus the amount of ammonium chloride, in a solution can be estimated. With regard to volumetric analysis two methods can be employed, either with or without indicator. The indicator used in this case is a solution of potassium chromate [$K_2CrO_4$]. This solution gives a red precipitate of silver chromate with a solution of silver nitrate—$K_2CrO_4 + 2AgNO_3 = Ag_2CrO_4 + 2KNO_3$.

If a few drops of $K_2CrO_4$ are added to a solution of ammonium chloride, and then silver nitrate added drop by drop, it will be noticed that a red colour is produced which disappears on shaking, leaving a white precipitate. Thus a point can finally be reached where a permanent red precipitate is obtained. It is thus evident that the silver nitrate first precipitates the silver chloride, and then the silver chromate. Potassium chromate can therefore be used to indicate the end of the first reaction, namely, the precipitation of the chloride.

NOTE.—(*a*) Acids must not be present, as silver chromate dissolves in dilute acids.

(*b*) Silver chloride is insoluble in acids, but soluble in $NH_4OH$.

(*c*) $\frac{N}{10}$ $AgNO_3$ contains 16·997 gms. per litre, or 1 c.c. = 0·016997 gm. of $AgNO_3$, and is equivalent to 0·003545 gm. of Cl.

*Method* (*a*), *without using Indicator.*—Take 20 c.c. of the ammonium chloride solution and place in a well-stoppered glass bottle. Then run in the $\frac{N}{10}$ $AgNO_3$ from your burette, shaking well after every few drops. The shaking helps the precipitate to coagulate [collect together], and it then settles to the bottom of the solution in the bottle. Continue this process until no further precipitate is produced when a drop of silver nitrate is added. Now, 1 c.c. of $\frac{N}{10}$ $AgNO_3$ is equivalent to 0·003545 gm. of Cl, *i.e.* to 0·00535 gm. $NH_4Cl$ [see equation, p. 52]. The strength of the ammonium chloride is thus readily calculated.

*Method* (*b*), *using* $K_2CrO_4$ *as Indicator.*—Again take 20 c.c. of the ammonium chloride solution and place in a white porcelain dish. Add two or three drops of $K_2CrO_4$ solution and run in $\frac{N}{10}$ $AgNO_3$ until a permanent colour is obtained. It is better to stir the solution in the basin with a glass rod during the titration. Calculation is the same as before.

*Questions B.*—(1) Which method do you consider more accurate, (*a*) or (*b*)? Give reasons in support of your choice.

(2) What sources of error do you think occur?

(3) Why do you think it is suggested you should use a white porcelain dish for the titrations in method (*b*)?

(4) Can you suggest any reason for stirring the solution in the basin in method (*b*)?

**78.** Find the strength of a barium chloride solution [in grams per litre] using $\frac{N}{10}$ $AgNO_3$.

*Method.*—In this experiment $K_2CrO_4$ cannot be used as an indicator because barium chromate would be formed, and this is insoluble in acids. Therefore, take 20 c.c. of the solution of barium chloride and place in a stoppered bottle and titrate as in method (*a*) for the estimation of ammonium chloride (Exp. 77). The equation is as follows :—

$$\begin{array}{llll} BaCl_2 + & 2AgNO_3 = & 2AgCl + & Ba(NO_3)_2 \\ 210 & [169{\cdot}9 \times 2] & 2 \times 143{\cdot}38 & \end{array}$$

### Section IX.—Preparations.

**79.** Prepare pure sodium chloride.

*Method.* — Make a saturated solution of table salt in a beaker. Pass in HCl until no more salt is precipitated. Filter, wash the salt ~~solution~~ with hydrochloric acid [sol. of gas], and heat strongly.

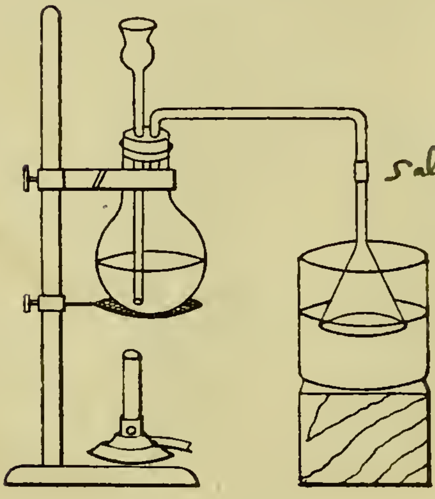

Fig. 29.

*Questions B.*—(1) Why does the salt separate ?

(2) What is the object of the funnel at the end of the delivery tube ?

(3) Why is HCl solution used for washing the salt ?

(4) Why is it good to heat strongly ?

(5) How would you test to see whether you had removed all the HCl ?

**80.**—Prepare 10 gms. of the neutral and acid salts of sulphuric acid.

(*a*) Neutral sodium sulphate—$Na_2SO_4$.

(*b*) Acid sodium sulphate—$NaHSO_4$.

*Questions A.*—(1) Write out your method.

(2) What are the solubilities of the two salts in water ?

(3) By what tests would you distinguish the two salts ?

**81.** Preparation of sodium thiosulphate.

*Question A.*—What is the formula for "thio" ? Why is it called by the name ?

*Method.*—Prepare 200 c.c. of cold saturated sodium carbonate solution. Take 100 c.c. and pass in sulphur dioxide until no more is absorbed. Add the other 100 c.c.

*Question.*—Write the equations for these two reactions.

Boil and add sulphur in small quantities at a time until no more is dissolved. Filter, evaporate to a quarter bulk, and then crystallise.

*Questions B.*—(1) Write the equation for the last change.
(2) Why do you evaporate to a quarter the bulk?

**82.** Prepare sodium bicarbonate from sodium carbonate.

(*a*) The normal carbonate is twice as soluble as the bicarbonate, hence devise your own method.

Test the solution of the bi-carbonate with litmus, methyl orange, and phenol phthalein.

Boil some bicarbonate solution and see whether $CO_2$ is evolved.

Heat some dry bicarbonate and see whether $CO_2$ is evolved.

(*b*) Prepare 10 gms. of sodium carbonate from the bicarbonate.

**83.** Prepare potassium nitrate from sodium nitrate and potassium chloride.

First look up the solubilities of all substances taking part.

Take 20 gms. $NaNO_3$, add the equivalent weight of KCl, and dissolve in 20 c.c. of boiling water. Filter off the salt undissolved, crystallize, and then dry and weigh the crystals.

*Questions B.*—(1) Is the nitre pure? Give full reasons for opinion.
(2) How can you ensure its purity?

**83a.** Prepare potassium chlorate as in the manufacture. (See special sheet.)

**84.** Prepare potash alum, $K_2SO_4,Al(SO_4)_3,24H_2O$.

*Method.*—Take 20 gms. of potassium sulphate, dissolve it in just sufficient cold water, and add an equivalent weight of aluminium sulphate [$12H_2O$], likewise dissolved in the smallest possible quantity of water. Allow to stand and crystallise.

**85.** Prepare pure ammonium chloride from gas-liquor.

*Method.*—Distil 100 c.c. of gas-liquor from a round-bottom flask. Collect the gas in 50 c.c. dilute HCl, evaporate to dryness. Sublime the dry salt by heating it in a dish over which a piece of perforated filter-paper is placed, covered by another dish as shown in figure 30.

**86.** Prepare metallic copper from azurite.

*Method.*—Dissolve the mineral in strong hydrochloric acid; evaporate off the excess of acid, and then add sheet iron to precipitate the copper. Collect the copper, wash with methylated spirits, and dry carefully and weigh. So form an idea of the percentage of copper in the ore.

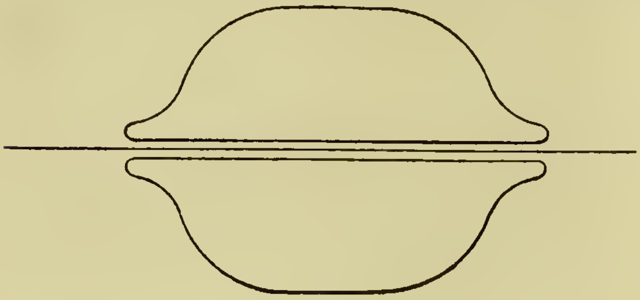

FIG. 30.

**87.** Prepare copper sulphate crystals [$CuSO_45H_2O$] from copper pyrites.

*Method.*—The powdered pyrites is heated in a basin in the fume cupboard with conc. nitric acid. This oxidises the sulphide to sulphate, which is then crystallised out.

**88.** Prepare 10 gms. copper carbonate from the sulphate by precipitation with sodium carbonate.

**89.** Prepare the nitrate from copper, and convert it into the oxide. Make 5 gms. of the oxide. From your results calculate the equivalent of copper.

**90.** Prepare cuprous chloride from copper, copper oxide, and hydrochloric acid.

*Method.*—Take 10 gms. of copper oxide, 20 gms. of copper, 25 c.c. strong hydrochloric acid. Heat on a sand bath for an hour, then pour into a litre of cold water. Allow the white precipitate to settle, wash with dilute hydrochloric acid, drain and wash with a little methylated spirits to dry rapidly.

**91.** Convert a 3*d.* piece [supplied by student] into silver chloride.

*Method.*—Dissolve it in nitric acid, and precipitate the silver with hydrochloric acid. Filter, dry, and weigh.

**92.** Pass coal gas over 1 gm. of the silver chloride obtained from 91, and weigh the silver obtained. Assuming the atomic weight of silver to be 108 ($H = 1$), calculate the atomic weight of chlorine.

**93.** Obtain silver by cupellation.

*Method.*—Wrap up the silver from Exp. 92 in pure lead foil and place in a cupel. Heat it in a muffle furnace until the lead is all oxidised away.

**94.** Obtain the metal from galena.

*Method.*—First heat the powdered sulphide in air to oxidise. Then mix with carbon and a little sodium carbonate, and heat strongly in a crucible.

**95.** Obtain litharge from lead carbonate.

*Method.*—Simply heat as gently as required.

**96.** Convert litharge into red lead.

*Method.*—Simply heat gently for at least an hour in air at a temperature of about dull red heat [400°].

**97.** Convert red lead into lead peroxide.

*Method.*—Add strong nitric acid to red lead. Lead peroxide will be left.

**98.** Prepare 5 gms. of lead chloride.

*Method.*—Add hydrochloric acid to lead nitrate solution. Collect and dry the precipitate, and crystallise from a little hot water.

**99.** Prepare 2 gms. of lead iodide.

*Method.*—Add potassium iodide to lead nitrate. Dissolve in hot water and cool slowly.

**100.** Obtain mercury from cinnabar.

*Method.*—Powder up 1 gm. of cinnabar; add dry sodium carbonate. Place in hard glass tube of shape shown in figure, and heat strongly. Collect the mercury in water; drain off the water, dry with filter-paper, and weigh the mercury.

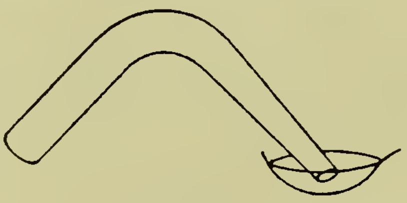

Fig. 31.

**101.** Prepare mercuric iodide.

*Method.*—Grind up the mercury obtained in 100 with a few crystals of iodine moistened with a drop of water.

**102.** Convert 2 gms. of zinc carbonate into the oxide, and calculate the purity of the zinc carbonate.

*Method.*—Heat the carbonate strongly.

**103.** Prepare zinc sulphate from the oxide.

*Method.*—Dissolve in dilute sulphuric acid and crystallise.

**104.** Prepare magnesium sulphate [$MgSO_47H_2O$] from magnesite.

*Method.*—Dissolve in sulphuric acid and crystallise.

**105.** Prepare ferrous ammonium sulphate [$FeSO_4$-$(NH_4)_2SO_47H_2O$].

*Method.*—Add molecular quantities of the two salts to make 30 gms. of the double salt.

35 gms. $FeSO_4 7H_2O$ in 60 c.c. of hot water [+ drop of $H_2SO_4$].

24 gms. $(NH_4)_2SO_4$ in 30 c.c. of hot water.

Filter each separately, mix, and cool. Filter, drain on a porous plate, and leave to dry.

## SECTION X.—Atomic Theory.

**106.** Object.—Find the density of air.

Fit up a half-litre round-bottom flask with a rubber bung, through which a tube passes, closed by a rubber tube and clip.

Place about 30 c.c. of water in the flask, and boil the water for five minutes to drive all the air out of the flask. Remove the flame and immediately clip the tube. Allow the flask to cool, then weigh it. Open the clip and weigh again.

*Questions A.*—(1) How will you find the volume of the air which has entered ?

(2) How will you calculate the weight of a litre of air from the experimental results ?

**107.** Object.—Find the density of carbon dioxide.

Dry a half-litre flask; fit it with a good cork, and then nearly fill it with carbon dioxide from a Kipps apparatus. Cork it and weigh it. Then open the flask under 1 per cent. caustic soda solution and absorb all the carbon dioxide, the alkali rising to take its place. The volume of alkali that enters the flask will be the volume of carbon dioxide that has been absorbed.

*Questions A.*—(1) How will you find the volume of carbon dioxide that entered ?

(2) How will you find the weight of carbon dioxide ?

**108.** Object.—Find the density of hydrochloric acid.

The experiment is practically the same as for carbonic acid.

*Question A.*—Write out the method you will adopt.

**109.** Object.—Find the density of ammonia.

This experiment only differs from the two previous ones in the use of 1 per cent. sulphuric acid instead of soda.

*Questions A.*—(1) Is there any advantage in using dilute acid ?

(2) Write out the method you will adopt, and show how you will calculate the density.

**110.** Object. Compare the rates of diffusion.

The apparatus consists of a small pipeclay plug, *p*, through which different gases can diffuse into the vacuum caused by inverting the mercury tube *t*. To carry out an experiment, the clip *b* is closed, and the tube *t* filled with mercury, closed with the finger, and then inverted as shown in the figure. Air is blown by means of a rubber bulb through a drying tube, then through the rubber tube *a*, and then out through the capillary tube *c*, which dips into an acid solution of potassium permanganate; the clip on *a* is closed, and the clip on *b* is opened. The air diffuses into the vacuum, and the coloured liquid rises in the capillary. The time required for the liquid to rise from $S_1$ to $S_2$ is taken, the experiment repeated until concordant results are obtained; then hydrogen and, later, carbon dioxide are substituted for air, and their times taken. Tabulate the times, the square of the times, and the densities of the gases.

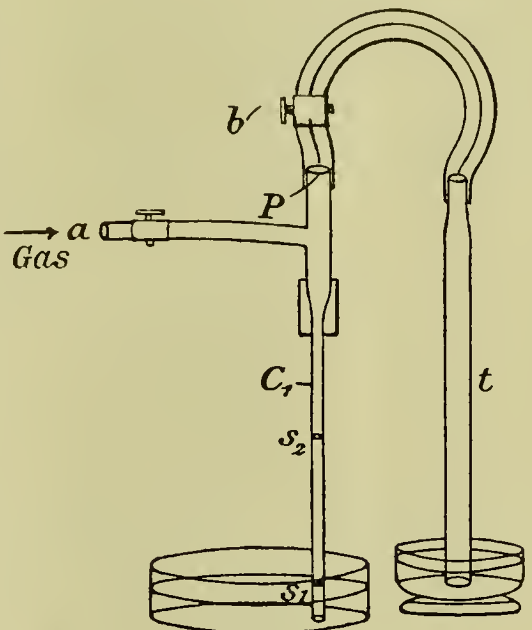

Fig. 32.

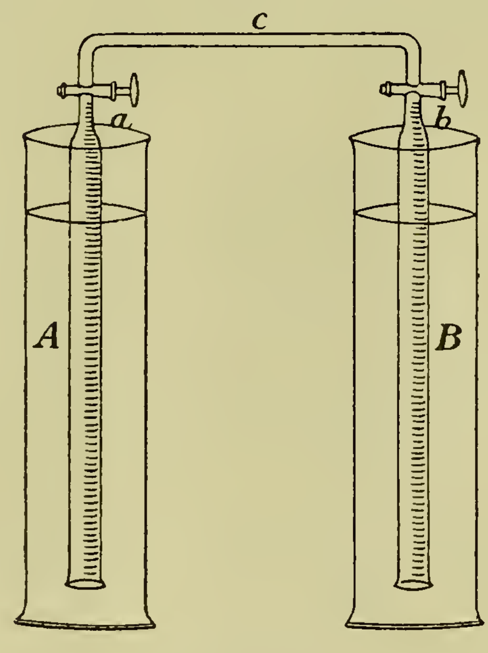

Fig. 33.

**111.** Object.—Find the composition of water by volume.

A known volume of hydrogen is mixed with a known volume of air and passed over warm platinised asbestos. Combination takes place and contraction ensues, from which the combining proportions can be calculated.

In *A* take about 20 c.c. of air, and bubble into *B* about 20 c.c. of hydrogen, using a Kipps apparatus. Measure the gases, after allowing them to remain in the tube five minutes to acquire the temperature of the water. Then pass nearly all the hydrogen from *B* into *A*; warm the asbestos tube *c* with a luminous Bunsen flame carefully, allow the mixed gases to pass into *B*, and continue to pass them backwards and forwards until no further change of volume appears to take place. Allow the gases to stand for five minutes again, read the volumes, add the two together, subtract the new volume from the original volume of the two gases to obtain the contraction. As all the oxygen will have been used up, it is easy to calculate how much hydrogen has combined with it.

*Question A.*—Set down the method of calculating.

**112.** Find the composition of hydrochloric acid by volume.

Fill a tube with dry hydrochloric acid gas, and stand it over mercury as in the figure. Measure the volume accurately at atmospheric pressure. Then introduce a small piece of sodium. Contraction will take place. Measure the new volume and the new pressure, and calculate what it would be at atmospheric pressure.

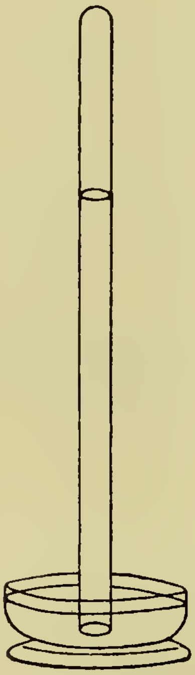

FIG. 34.

*Question A.*—Why is it not necessary to take account of the temperature?

*Question B.*—What do results prove as to the composition of hydrochloric acid?

**113.** Find the composition of carbon monoxide.

Pass pure carbon monoxide over copper oxide in a boat. The loss in weight of the copper oxide gives the weight of oxygen that combines with the carbon monoxide. Collect the carbon dioxide formed in weighed potash bulbs, and weigh again. The results will give the weight of oxygen and the weight of carbon dioxide. By difference we get the weight of carbon monoxide. So we have weight of oxygen, carbon monoxide, carbon dioxide.

*Question A.*—(1) Find the volumes of oxygen and carbon dioxide which have combined together by dividing each weight by the density of the corresponding gas. In the same way calculate the volume of carbon dioxide which has been produced.

**114.** Find the composition of ammonia by volume.

Fill the tube with chlorine, then run in a solution of ammonia drop by drop. The chlorine will combine with the hydrogen and liberate the nitrogen.

*Questions A.*—(1) Write down in detail how you will carry out the experiment.

(2) State clearly the reasoning by which you deduce the composition of ammonia.

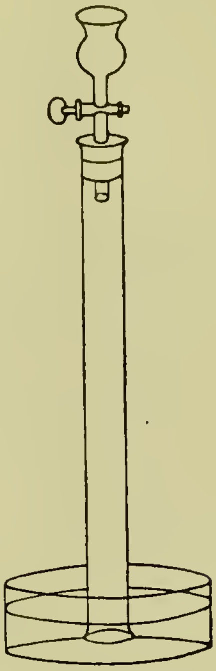

FIG. 35.

**115.** Find the atomic weight of potassium, chlorine, silver.

Ignite a weighed quantity of potassium chlorate very carefully, and find the loss in weight. Convert the potassium chloride so formed into silver chloride, and weigh again.

Reduce the silver chloride to silver in a current of hydrogen. Take the atomic weight of oxygen as 16, then from your results calculate—

(1) The molecular weight of potassium chloride [$M_1$].

(2) The molecular weight of silver chloride [$M_2$].

(3) The weight of silver in the molecular weight of silver chloride [X].

(4) The weight of chlorine in the molecular weight of silver chloride [Y].

(5) The weight of potassium in the molecular weight of potassium chloride [Z].

**116.** Find the vapour density of benzene by Victor Meyer's method [see special sheet].

**117.** Find the molecular weight of naphthalene by Raoult's method [see special sheet].

**118.** Find the molecular weight of naphthalene by Landsberger's method [see special sheet].

**119.** Find the electro-chemical equivalent of silver and copper [see special sheet].

**119*a*.** Find the specific heat of mercury.

Take about 60 gms. of mercury, place in a test-tube, and then put into boiling water for five minutes. Then pour into cold water contained in a calorimeter.

*Question A.*—Write out in detail how you intend to carry out the experiment.

*Question B.*—Using your value obtained by experiment for the specific heat of mercury, find by calculation the "atomic heat" of that element.

### Section XI.—Special Volumetric Analysis.

**120.** Object.—Find if the given substance is carbonate or bicarbonate of sodium.

*Question A.*—Write out your method before starting.

**121.** Object.—Find the weight of magnesium in the given tube.

*Method.*—Dissolve the magnesium in excess of standard acid, and then estimate the excess by means of caustic soda. By this means you can determine the amount of acid used up in dissolving the magnesium, and then from the equation representing the action which takes place find the amount of magnesium present.

**122.** Object.—Find the percentage of pure calcium chloride in the given example

*Question A.*—How will you weigh out the calcium chloride?

*Method.*—Take a weighed quantity, dissolve in water, and make up to a known volume. The calcium chloride may then be estimated by either of the methods adopted in Exp. 76 or with standard $AgNO_3$ without using an indicator.

**123.** Object.—Find the amount in grams per litre of sodium and barium chlorides in a solution.

*Method.*—First evaporate a known volume to dryness in a weighed dish, and so obtain the total solids present. The barium chloride may then be estimated by either of the methods used in Exp. 76, and the sodium chloride obtained by difference.

*Question A.*—Is there any other way of analysing this solution?

**124.** Object.—Find the amount in grams per litre of ammonium and sodium hydrates in a solution.

*Method.*—First find the total alkali present by titration with standard acid, then boil off the ammonia, and estimate the caustic soda remaining with standard acid. The ammonium hydrate is then obtained by difference.

*Question B.*—Describe another method of doing this experiment.

**125.** Object.—Find the percentage amount of caustic soda and sodium carbonate in commercial caustic soda.

*Method.*—First determine the total alkali present by titration with standard acid, then precipitate the carbonate by the addition of barium chloride solution. Add the $BaCl_2$ solution very carefully so as not to use excess [add until no further precipitate forms]. Allow the precipitate to settle, and then titrate measured quantities of the clear liquid with standard acid, and so obtain the amount of sodium hydrate. The carbonate is got by difference.

*Questions B.*—(1) What is the theory of the above estimation?

(2) Why must you be careful not to add excess of $BaCl_2$?

(3) Can you suggest any sources of error in this estimation?

*N.B.*—Alternate method, using the two indicators methyl orange and phenol phthalein.

**126.** Object.—Standardise a solution of potassium permanganate with ferrous ammonium sulphate.

*Preliminary Considerations.*—When sulphuric acid acts upon potassium permanganate [$K_2Mn_2O_8$] the following reaction tends to take place:—

$$\underset{316{\cdot}5}{K_2Mn_2O_8} + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 3H_2O + \underset{80}{5O}$$

No evolution of oxygen takes place as long as the solutions are cold and dilute. If certain oxidisable substances are present, however, the reaction indicated above takes place until the permanganate is all decomposed or the oxidisable body is completely oxidised. For example, ferrous sulphate is oxidised to ferric sulphate—

$$\underset{2(55{\cdot}9 + 32 + 64)}{2FeSO_4} + H_2SO_4 + O_{16} = Fe_2(SO_4)_3 + H_2O$$

To demonstrate these reactions, prepare a solution of ferrous sulphate in water, acidify with dilute $H_2SO_4$, and add to it drop by drop a solution of potassium permanganate, with constant shaking of the liquids. The permanganate solution is decolorised, and this continues until all the ferrous sulphate is oxidised. The deep tint of the permanganate then remains permanent, indicating the end of the reaction. It is now evident how potassium permanganate solution can be applied to the determination of iron [and other substances]. The permanganate acts as its own indicator. The standard solution of potassium permanganate is prepared on the oxygen basis. A *normal* solution contains in 1 litre enough salt to give one equivalent of oxygen [8 gms.]. From the above equation it will be seen that it is 315·5 ÷ 10 = 31·65 gms. It is usual to employ a $\frac{N}{10}$ solution; this contains 3·165 gms. of permanganate per litre, and corresponds to 0·0008 gm. of oxygen, or to 0·00559 gm. of ferrous iron [see the second equation given above]. The complete equations representing the reactions between ferrous sulphate and potassium permanganate are as follows:—

(*a*) $K_2Mn_2O_8 + 3H_2SO_4 = K_2SO_4 + 2MnSO_4 + 3H_2O + 5O.$
(*b*) $10FeSO_4 + 5H_2SO_4 + 5O = 5Fe_2(SO_4)_3 + 5H_2O.$

Combined in one equation—

$$K_2Mn_2O_8 + 10FeSO_4 + 8H_2SO_4 = 5Fe_2(SO_4)_3 + K_2SO_4 + 2MnSO_4 + 8H_2O.$$

The following points should be noticed in connection with the use of potassium permanganate:—

(*a*) Ferrous iron is only indicated in titrations with it.

(*b*) A sufficiency of sulphuric acid must be present. Even at high temperatures dilute $H_2SO_4$ exerts no prejudicial effect.

(*c*) HCl must not be present unless very dilute, and the temperature must even then be kept low.

(*d*) The potassium permanganate solution acts upon indiarubber, and should not be kept in contact for any length of time with rubber tubing.

*Question A.*—(1) Can you give a reason why HCl must not be present with permanganate unless very dilute, and the temperature even then having to be kept low?

*Method* of standardisation of $K_2Mn_2O_8$ with ferrous ammonium sulphate—

$$[FeSO_4(NH_4)_2SO_46H_2O].$$
$$392{\cdot}26.$$

Weigh out about 5 gms. of pure, dry ferrous ammonium sulphate and dissolve in distilled water [which has been previously boiled and cooled rapidly], and make up to 100 c.c. Take 20 c.c. of this solution and titrate with the given $K_2Mn_2O_8$ solution. Ferrous ammonium sulphate contains $\frac{1}{7}$ its weight of iron. The calculation is therefore perfectly straightforward. It might be mentioned that this substance acts like $FeSO_4$, the ammonium sulphate making no difference to the reaction.

*Question B.*—Why are you told to use boiled-out distilled water for dissolving the ferrous ammonium sulphate?

127. Object.—Find the percentage of iron in a sample of iron wire.

*Apparatus.*—Fit up the apparatus shown.

*Method.*—Take a piece of clean iron wire (about 1 grm.) and weigh accurately. Then place in flask *A* along with it about 100 c.c. of dilute sulphuric acid [a few small crystals of $Na_2CO_3$ may be placed in the flask so as to drive out all the air]. The water in the beaker *B* should be distilled water which has been previously boiled and cooled rapidly. This acts as a trap, and prevents oxygen from getting into the flask *A*. Now gently warm the contents of *A* until all the iron has dissolved, and then rapidly transfer the contents of *A* into a 250 c.c. measuring-flask, and make up to the mark with distilled water which has been previously boiled and cooled rapidly. Now titrate measured quantities with the standard permanganate, and so obtain the amount of iron present in solution.

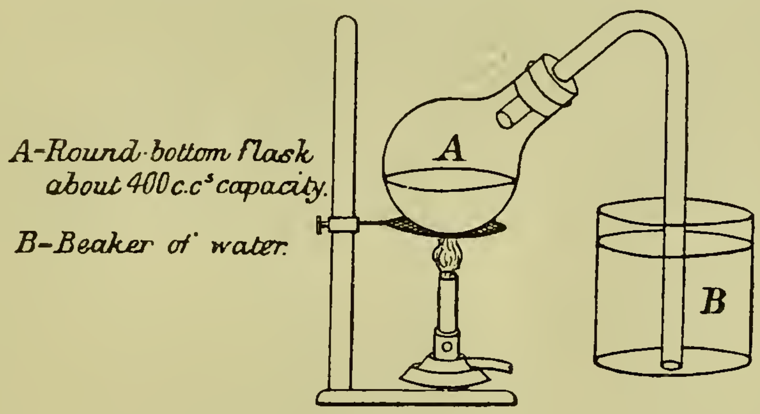

Fig. 36.

**128.** Object.—Find the percentage of iron present in a sample of spathic iron ore by means of standard permanganate.

Note.—The iron is present in the ferrous and ferric states. The amount of ferrous iron is first estimated, and then the ferric iron is reduced to the ferrous state and the total amount of iron estimated.

*Method.*—Weigh out accurately about 2·5 gms. of the finely powdered iron ore. Heat this in a covered beaker over a small flame for about half an hour with hydrochloric acid diluted with its own volume of boiled-out water. Dilute this solution slightly with boiled-out water, allow the undissolved matter to subside, and decant the liquid. Then treat the residue with a small quantity of the dilute acid, and decant the liquid. This procedure will usually have extracted all the iron from the ore. The residue should, however, be treated with the acid until the solution gives no colour with potassium thiocyanate solution. Wash the residue well with distilled water, using as small a quantity of water as possible, and then mix the main solution and washings together and make up to 250 c.c. with boiled-out water [distilled]. Now titrate measured quantities with the standard permanganate solution. This gives the ferrous iron.

*Reduction of Ferric Iron with Stannous Chloride.*—50 c.c. of the iron solution are placed in an 8-oz. flask, 2 c.c. of strong HCl are then added, and the liquid heated to boiling. Clean, freshly made solution of stannous chloride in HCl, diluted with half its own volume of boiled distilled water, is next added from a pipette drop by drop until the yellow colour of the iron solution just disappears. About 6 c.c. of a saturated solution of mercuric chloride solution are then added. This *must* produce turbidity in the liquid, indicating that the stannous chloride solution has been added in excess. The solution is then cooled and titrated with the standard permanganate. This gives the total ferrous iron after reduction.

*Questions B.*—(1) Do you think this is an accurate method for estimating the iron in this ore? Give reasons in support of your answer?

(2) Can you suggest any other reducing agents which could be used instead of stannous chloride?

129. Object.—(*a*) Find the percentage of iron in a sample of iron wire; and (*b*) find the percentage of iron in spathic iron ore, using standard bichromate solution.

*Apparatus.*—The same apparatus will be required as in the previous experiments.

*Preliminary Considerations.*—When sulphuric acid acts upon potassium bichromate [$K_2Cr_2O_7$] the following change takes place:—

$$\underset{294\cdot5}{K_2Cr_2O_7} + 4H_2SO_4 = K_2SO_4 + Cr_2(SO_4)_3 + H_2O + \underset{48}{3O}$$

As in the case of permanganate [see p. 63], no evolution of oxygen takes place when the solutions are cold and dilute. Take a solution of ferrous sulphate and acidulate with dilute $H_2SO_4$; then add a solution of potassium bichromate drop by drop, constantly shaking the liquids. The green colour of chromium sulphate [$Cr_2(SO_4)_3$] soon indicates the progress of the reaction; but, unlike permanganate, the end of the reaction cannot be determined by this colour-change. In this case an "external indicator" must be employed. To illustrate this, prepare a dilute solution of potassium ferricyanide [$K_3Fe(CN)_6$] by shaking a crystal of the substance with cold water. A faintly coloured solution is most suitable. Test this solution with ferrous sulphate solution and with a solution of a ferric salt [ferric chloride is most convenient]. Notice that the ferrous salt gives a dark blue colour or precipitate, and the ferric salt a dark brown coloration. Now, by means of a glass rod, place a series of drops of the dilute potassium ferricyanide solution on a white tile. To a solution of ferrous sulphate acidulated with dilute $H_2SO_4$ add $K_2Cr_2O_7$ solution with constant shaking, and from time to time take out a drop of the liquid on the end of a glass rod and touch it on a spot of ferricyanide solution. As long as unoxidised ferrous sulphate remains in the solution, a blue colour will form; thus the colour ceases to appear when all the iron is in the ferric state. Potassium ferricyanide is thus available as an "external indicator" to indicate the end point of a titration of ferrous iron with potassium bichromate. Standard bichromate is prepared on the oxygen basis, and from the equation above it will be seen that the quantity of bichromate equivalent to 8 gms. of oxygen is

$\frac{294{\cdot}5}{0{\cdot}6} = 49{\cdot}083$ gms. Therefore a $\frac{N}{10}$ bichromate solution contains 4·9083 gms. per litre; therefore 1 c.c. is equivalent to 0·0008 gm. of oxygen and 0·00559 gm. of Fe. The complete equations representing the reaction between ferrous sulphate and potassium bichromate are—

(*a*) $K_2Cr_2O_4 + 4H_2SO_4 = K_2SO_4 + Cr_2(SO_4)_3 + 4H_2O + 3O.$
(*b*) $6FeSO_4 + 3H_2SO_4 + 3O = 3\ Fe_2\ (SO_4)_3 + 3H_2O.$

Combined in one equation—

$$K_2Cr_2O_7 + 6FeSO_4 + 7H_2SO_4 = 3Fe_2(SO_4)_3 + K_2SO_4 + Cr_2(SO_4)_3 + 7H_2O.$$

The same points should be noticed in the case of bichromate as with $K_2Mn_2O$ [see p. 64]. It may be noted that HCl is not so prejudicial in this case as with permanganate.

*Method.*—(*a*) Proceed as in Exp. 127, except that you use potassium bichromate for the titrations and potassium ferricyanide as indicator.

(*b*) Again proceed exactly as in Exp. 128.

*Questions B.*—(1) Do you think this is a more accurate method of estimation then using permanganate solution—(*a*) In estimating iron in iron wire? (*b*) In estimating iron in spathic iron ore?

(2) Suppose you were asked to find the amount of iron in a sample of ferrous sulphate, which solution would you use, permanganate or bichromate? Give reasons for your choice.

**130.** Object.—Make a decinormal solution of iodine, and then standardise a solution of sodium thiosulphate.

*Preliminary Considerations.*—Dissolve a little iodine in a solution of potassium iodide [KI], and add to it some sulphurous acid. The iodine is gradually decolorised. The action is represented by the following equation :—

$$\begin{array}{l} I_2 + H_2O + H_2SO_3 = 2HI + H_2SO_4 \\ 126{\cdot}97 \times 2 \end{array}$$

This gives a method of estimating sulphur dioxide in solution. Add a solution of iodine of known strength to the sulphurous acid solution until the colour of the iodine remains permanent. When using iodine solution instead of using the iodine itself to indicate the end point, a solution of starch is used as an indicator.

*Preparation of Starch Solution.*—Make about 1 gm. of starch into a paste with cold water, and then pour into about 150 c.c. of boiling water. Boil for about five minutes longer, and then allow to settle. Pour off the clear liquid.

Add a little starch solution to about 6 c.c. of water in a test-tube, and then add a solution of iodine in potassium iodide. Note the blue colour produced. Now warm the solution. Note that the colour disappears. Cool under the tap, and notice that the blue colour returns. Then add a little sulphurous acid, and the colour will again disappear. Sodium hydrate destroys the blue colour, but mineral acids and iodides have no action on it. Like $K_2Cr_2O_7$, standard solutions of iodine are made on the oxygen basis. $\frac{N}{10}$ solutions are generally used, and from the equation on the previous page it will be seen that these contain 12·697 gms. of iodine per litre.

(*a*) *Preparation of* $\frac{N}{10}$ *Iodine.*—Weigh out 6·349 gms. of pure iodine [this will be supplied], and dissolve in about 250 c.c. of cold distilled water containing 9 gms. of potassium iodide. When all iodine has dissolved, make up to 500 c.c.

(*b*) *Standardisation of a Solution of Sodium Thiosulphate.*—The reaction between iodine and sodium thiosulphate is represented by the following equation :—

$$\begin{aligned}&2Na_2S_2O_3 + I_2 = 2NaI + Na_2S_4O_6\\&316{\cdot}44 + 253{\cdot}94\end{aligned}$$

Sodium thiosulphate contains 5 molecules of water of crystallisation, therefore its molecular weight is 248·3 ; and since 1 molecule corresponds to 1 molecule of iodine [see equation], a solution containing 24·83 gms. per litre is decinormal. To titrate the thiosulphate against the iodine, take a measured quantity of the latter and run in the thiosulphate solution until the colour of the iodine has nearly disappeared. Then add a little starch solution, and continue adding the thiosulphate until the blue colour disappears. This is the end point of the reaction. [The solution of the iodine should be well shaken frequently during the time the thiosulphate is being added.] From the above equation the strength of the above solution can be calculated. [Don't overlook the water of crystallisation.]

*Question B.*—Can you give any reason why the starch solution is not added until the colour of the iodine has nearly disappeared?

**131.** Object.—Estimate the available chlorine in bleaching powder.

*Question A.*—Show how to obtain the theoretical value of "available chlorine" in bleaching powder.

*Method.*—In this experiment the bleaching powder is made to give up its chlorine by the addition of hydrochloric acid, and the chlorine produced is used in turning out iodine from a solution of potassium iodide. The iodine is then estimated with the $\frac{N}{10}$ thiosulphate solution. Weigh out 10 gms. of bleaching powder, place it in a smooth porcelain mortar, preferably one which is glazed inside, add a few c.c. of water. Rub the mixture into a paste by means of the pestle; then add more water, and continue the rubbing until a thin cream is formed; allow the solution to subside, and decant the supernatant liquid into a litre flask. Then treat the residue repeatedly with fresh quantities of water, stirring up and pouring off after every addition until the whole of the solid has been transferred to the flask. Finally, fill up the flask to the mark with water, and mix its contents well by shaking. Each c.c. of the solution will contain 0·01 gm. of bleaching powder. Take 25 c.c. of the above solution in a flask, and add excess of a solution of potassium iodide, acidifying the liquid with dilute hydrochloric acid. Then determine the amount of iodine liberated with sodium thiosulphate and starch. The amount of available chlorine in the bleaching powder is chemically equivalent to the iodine liberated.

**132.** Object.—Find the percentage of manganese dioxide [$MnO_2$] in a given sample.

*Apparatus.*—Fix up the apparatus as shown in the diagram.

A small round-bottom flask, *A*, of about 2 ozs. capacity connected to a tube, *B*, by means of a rubber joint. Upon the end of this is fused a thinner tube, *C*, which is bent as shown in the diagram, and is fitted to the bulb **U**-tube *D* by means of a rubber cork. The **U**-tube is immersed in water.

[NOTE. — If necessary, a second plain **U**-tube may be connected to *D*.]

*Question A.*—Why is the tube *C* fused to *B*, and not connected with rubber tubing?

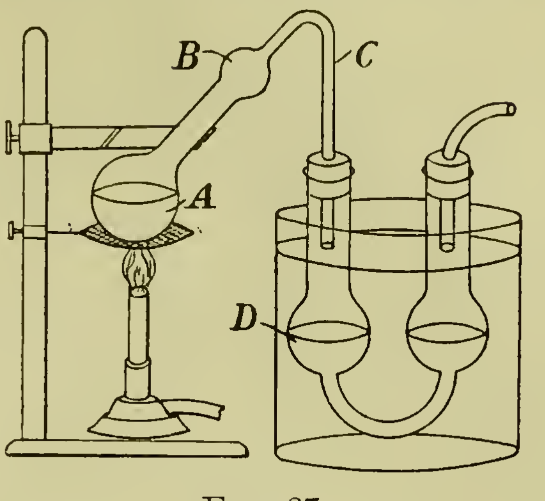

FIG. 37.

*Method.* — When manganese dioxide is heated with hydrochloric acid, chlorine gas is evolved. This gas is conducted into an excess of potassium iodide solution, and the iodine set free is estimated by means of sodium thiosulphate and starch solutions.

From the equation it is seen that 87·05 parts of $MnO_2$ can liberate 70·9 parts of chlorine and 253·72 parts of iodine.

$$\underset{87 \cdot 05}{MnO_2} + 4HCl = MnCl_2 + 2H_2O + \underset{70 \cdot 9.}{Cl_2.}$$

Weigh out accurately about 0·5 gm. of finely powdered manganese dioxide into the small flask. Half fill the bulbs of the **U**-tube *D* with strong potassium iodide solution. [If a second tube is used, this also contains potassium iodide solution.] Ascertain that the apparatus is air-tight, and then add about 200 c.c. of strong hydrochloric acid to the manganese dioxide in the flask. It will be found convenient to introduce a small piece of magnesite in the flask *A*. Now heat the flask gently by means of a small flame, and as soon as the manganese dioxide has dissolved, boil the acid until all the chlorine has been expelled. When all the chlorine has been passed into the absorption tube, pour out the contents into a beaker, and rinse out the tube also into the beaker. [If a second tube has been used and the contents are yellow, these also must be transferred to the beaker; if colourless, they can be neglected.] The free iodine is then at once titrated by means of sodium thiosulphate solution and starch. The free iodine thus found measures the chlorine liberated by the manganese dioxide, and two atoms of chlorine correspond to one molecule of $MnO_2$, as has been already explained.

*Questions B.*—(1) How did you test the apparatus to see if it was air-tight?

(2) Why are you asked to add a crystal of magnesite to the contents of *A*?

**133.** Object.—Find the amount of potassium iodide and chloride in a solution.

*Method.*—In this experiment you are told the total amount of solids present. Titrate with $\frac{N}{10}AgNO_3$, and so obtain the total halogens present. The amount of potassium iodide and chloride present are then readily obtained by calculation.

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